

Prepared in cooperation with the Triangle Area Water Supply Monitoring Project  
Steering Committee

# **Triangle Area Water Supply Monitoring Project, October 1988 through September 2001, North Carolina—Description of the Water-Quality Network, Sampling and Analysis Methods, and Quality-Assurance Practices**



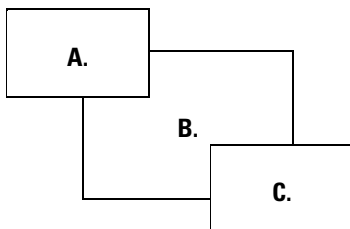
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**Cover photographs** (from U.S. Geological Survey, Raleigh Field Office files)

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- A.** Neuse River at Falls Lake dam
- B.** Lake Michie dam and tailrace
- C.** Deep River near Moncure, N.C.

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By Carolyn J. Oblinger

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U.S. Geological Survey**

**U.S. Department of the Interior**  
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## Conversion Factors, Definitions, and Acronyms and Abbreviations

Multiply	By	To obtain
<b>Length</b>		
foot (ft)	0.3048	meter (m)
mile (mi)	1.609	kilometer (km)
<b>Area</b>		
acre	0.4047	hectare (ha)
square mile (mi <sup>2</sup> )	2.590	square kilometer (km <sup>2</sup> )
<b>Volume</b>		
cubic foot (ft <sup>3</sup> )	0.02832	cubic meter (m <sup>3</sup> )
<b>Flow rate</b>		
acre-foot per day (acre-ft/d)	0.01427	cubic meter per second (m <sup>3</sup> /s)
acre-foot per year (acre-ft/yr)	1,233	cubic meter per year (m <sup>3</sup> /yr)
acre-foot per year (acre-ft/yr)	0.001233	cubic hectometer per year (hm <sup>3</sup> /yr)
foot per second (ft/s)	0.3048	meter per second (m/s)
cubic foot per second (ft <sup>3</sup> /s)	0.02832	cubic meter per second (m <sup>3</sup> /s)
million gallons per day (Mgal/d)	0.04381	cubic meter per second (m <sup>3</sup> /s)
<b>Mass</b>		
pound, avoirdupois (lb)	0.4536	kilogram (kg)

**Horizontal coordinate information** is referenced to the North American Datum of 1983 (NAD 83).

**Temperature** in degrees Celsius (°C) can be converted to degrees Fahrenheit (°F) as follows:

$$^{\circ}\text{F} = (1.8 \times ^{\circ}\text{C}) + 32$$

**Specific conductance** is given in microsiemens per centimeter at 25 degrees Celsius (μS/cm at 25 °C).

**Concentrations of chemical constituents in water** are given in this report either in milligrams per liter (mg/L) or micrograms per liter (μg/L). Liter (L) is a metric unit of volume equal to 2.1134 liquid pints.

**Water year**, as referred to in this report, is the period October 1 to September 30 and is identified by the calendar year in which the period ends.

## Acronyms and Abbreviations

DWQ	North Carolina Division of Water Quality
ECD	electron capture detector
EWI	equal-width increment
FID	flame ionization detector
FISP	Federal Interagency Sedimentation Project
FPD	flame photometric detector
GC	gas chromatography
GC/FID	gas chromatography with flame ionization detector
HPLC	high-performance liquid chromatography
IBSP	Inorganic Blind Sample Project
mL	milliliter
mm	millimeter
MPV	most probable value
MS	mass spectrometry
NDWRF	North Durham Water Reclamation Facility
NWIS	USGS National Water Information System
NWQL	National Water Quality Laboratory
OBSP	Organic Blind Sample Project
OWASA	Orange Water and Sewer Authority
PCB	polychlorinated biphenyls
PCN	polychlorinated naphthalene
QAS	Quality Assurance Section of the NWQL
TJCOG	Triangle J Council of Governments
USGS	U.S. Geological Survey

# **Triangle Area Water Supply Monitoring Project, October 1988 through September 2001, North Carolina—Description of the Water-Quality Network, Sampling and Analysis Methods, and Quality-Assurance Practices**

By Carolyn J. Oblinger

## **Abstract**

The Triangle Area Water Supply Monitoring Project was initiated in October 1988 to provide long-term water-quality data for six area water-supply reservoirs and their tributaries. In addition, the project provides data that can be used to determine the effectiveness of large-scale changes in water-resource management practices, document differences in water quality among water-supply types (large multiuse reservoir, small reservoir, run-of-river), and tributary-loading and in-lake data for water-quality modeling of Falls and Jordan Lakes. By September 2001, the project had progressed in four phases and included as many as 34 sites (in 1991). Most sites were sampled and analyzed by the U.S. Geological Survey. Some sites were already a part of the North Carolina Division of Water Quality statewide ambient water-quality monitoring network and were sampled by the Division of Water Quality. The network has provided data on streamflow, physical properties, and concentrations of nutrients, major ions, metals, trace elements, chlorophyll, total organic carbon, suspended sediment, and selected synthetic organic compounds.

Project quality-assurance activities include written procedures for sample collection, record management and archive, collection of field quality-control samples (blank samples and replicate samples), and monitoring the quality of field supplies. In addition to project quality-assurance activities, the quality of laboratory analyses was assessed through laboratory quality-assurance practices and an independent laboratory quality-control assessment provided by the U.S. Geological Survey Branch of Quality Systems through the Blind Inorganic Sample Project and the Organic Blind Sample Project.

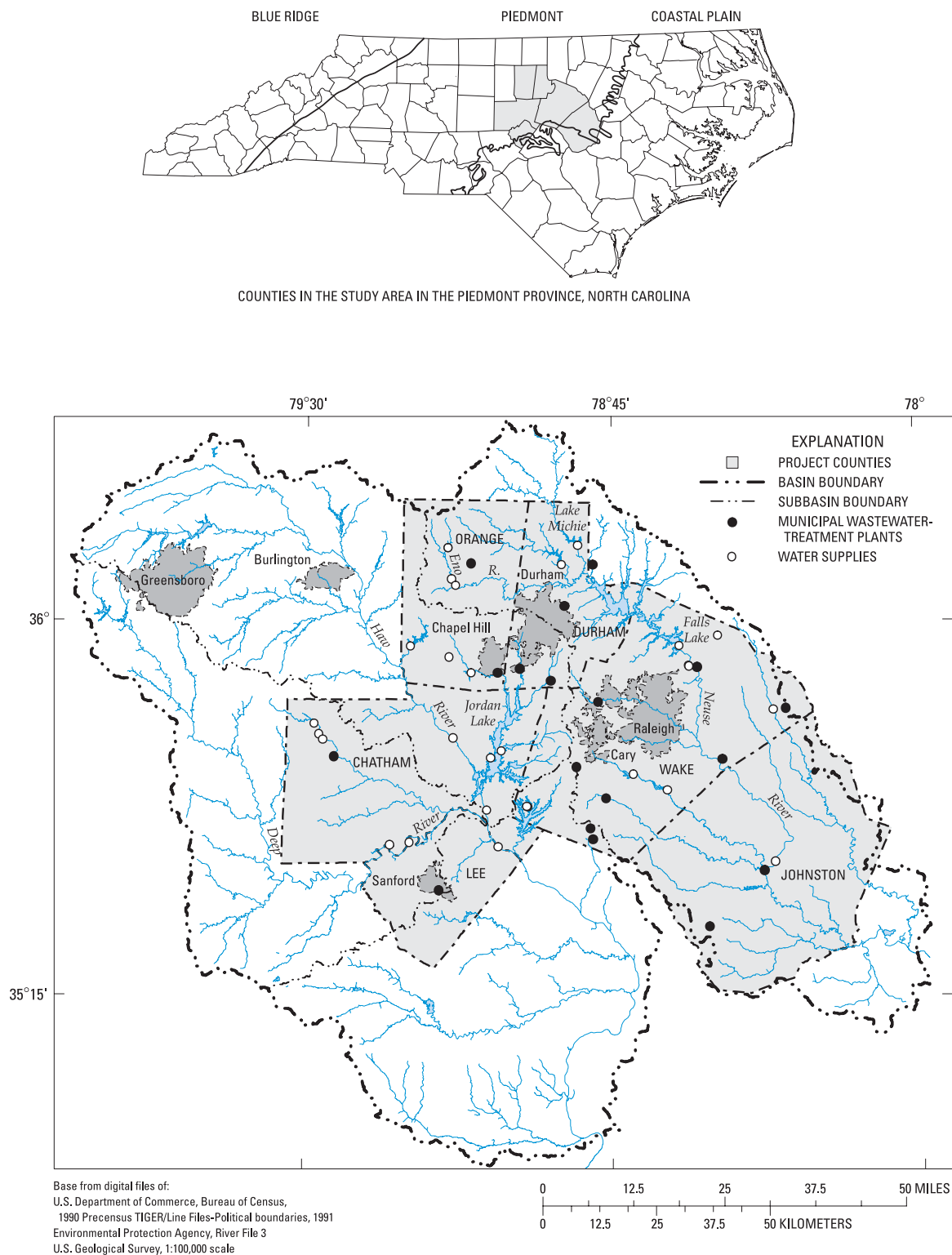
## **Introduction**

The Triangle Area Water Supply Monitoring Project was initiated in October 1988 in response to concern about the effects of increasing growth in the Triangle Area of North Carolina on the quality of drinking-water resources (fig. 1). The Triangle Area in the central North Carolina Piedmont includes the municipalities of Raleigh, Cary, Durham, and Chapel Hill. In 1988, two impoundment projects had been completed to provide new drinking-water supplies in the Triangle Area—the B. Everett Jordan and Falls of the Neuse Reservoirs (hereafter referred to as Jordan Lake and Falls Lake, respectively), and it was recognized that water supplies must be protected. The initial goal of the project was to collect water-quality data for six water-supply reservoirs and their tributaries in the Triangle Area. Some tributaries also are run-of-river water supplies. These water supplies were selected because they provide about 95 percent of the public surface-water supply for the area.

When initiated, the project objectives were to

- Expand the existing database for synthetic-organic chemicals and supplement the existing nutrient and trace-metal database so that long-term trends can be detected in major water supplies;
- Provide data that can be used to determine if large-scale changes in management practices are effective in producing improvements in water quality;
- Document spatial differences among water supplies and differences in water quality based on the source of water (large multiuse reservoir, small reservoir, and run-of-river); and
- Provide tributary loading and in-lake data for predictive water-quality models for Falls and Jordan Lakes.

## 2 Triangle Area Water Supply Monitoring Project, October 1988 through September 2001, North Carolina



**Figure 1.** Triangle Area Water Supply Monitoring Project study area in North Carolina and locations of major municipal water supplies and wastewater-treatment facilities.

Funding for the project has been provided by a consortium of municipalities and organizations with interest in drinking-water quality (table 1) and by the USGS. The consortium, which provides 50 percent of the project funding, was formally organized through an interagency agreement as the Triangle Area Water Supply Monitoring Project Steering Committee (hereafter referred to as the Steering Committee). The Triangle J Council of Governments (TJCOG) manages the project on behalf of the Steering Committee. The USGS provides the remaining 50 percent of project funding.

**Table 1.** Municipalities and organizations providing funding and membership to the Triangle Area Water Supply Monitoring Project Steering Committee in North Carolina.

Participants (locations shown in fig. 2)	Period of participation through 2002
Town of Apex	1988 – continuing
Town of Cary	1988 – continuing
Chatham County	1988 – continuing
City of Durham	1988 – continuing
Orange County	1988 – continuing
OWASA <sup>a</sup> (Towns of Chapel Hill and Carrboro)	1988 – continuing
Town of Hillsborough	1988 – continuing
Town of Pittsboro	1988 – 1992
City of Raleigh	1988 – 1995
Town of Sanford	1988 – 1999
Town of Smithfield	1988 – 1995

<sup>a</sup>Orange (County) Water and Sewer Authority.

Included in the study were sites that were routinely sampled by the North Carolina Department of Environment and Natural Resources, Division of Water Quality (DWQ), as part of the statewide ambient water-quality monitoring network. The DWQ sites selected for inclusion in the project network generally were located downstream from municipal wastewater discharges on tributaries to the area's raw-water supplies (table 2; fig. 2). To complete the Triangle Area Water-Supply Monitoring Project network, additional sites were established on lake and run-of-river raw-water supplies and their tributaries. These sites are sampled by the U.S. Geological Survey (USGS).

Expectations expressed in 1988 about growth in the Triangle Area have been realized. Based on census records, the population of the Metropolitan Statistical Area, defined by the U.S. Office of Management and Budget as Chatham, Durham, Franklin, Johnston, Orange, and Wake Counties, was about 860,000 in 1990 (North Carolina State Data Center, 2002a, b). Between 1990 and 2000, the area grew by 38 percent. Between 2000 and 2010, the area is projected to grow by another 29 percent, bringing the total population of the area to about

1,540,000. As a consequence of population growth, land use in the Triangle Area has changed from forest and agriculture to urban and suburban.

## Purpose and Scope

This report documents the history of the first four phases of the Triangle Area Water Supply Monitoring Project, including the scope, data-collection methods, laboratory analytical methods, and quality-assurance practices. The report documents sample-collection and processing methods for samples collected by the USGS and documents sample-analysis methods for samples analyzed by the USGS National Water Quality Laboratory. Summaries of stream- and lake-sample analyses (Appendixes A1, A2) and analyses of quality-control samples collected between October 1988 and September 2001 are presented. Quality-control data include blank samples and sample replicates that were collected to monitor the potential contamination of samples from such factors as sampling technique, sampling equipment and cleaning techniques, sample processing, and sample transport and shipping. A database containing USGS environmental and quality-control data and DWQ water-quality data collected from October 1988 through September 2001 also is included (Appendix A3; appendix tables are on a compact disc (CD) inside the back cover).

## Project History

The project has consisted of four phases since beginning in October 1988. The basic objectives for each phase are those listed in the Introduction. In addition to these objectives, each phase had a unique supplemental area of focus for water quality. A description of each phase, including the supplemental work unique to each phase, follows. In all phases, the North Carolina DWQ collected samples at DWQ ambient water-quality monitoring sites within the network according to the frequency established for the State program. In phase I, the DWQ (then named the Division of Environmental Management) also sampled Falls and Jordan Lakes.

The first phase lasted for 3 years, from October 1988 through September 1991 (table 3). The sampling network consisted of 31 sites (table 2). Of these, 9 sites were located near stream or lake water supplies, 7 were located below wastewater discharges, and 15 were either lake or tributary sites (table 2). In general, the USGS sampled the tributary sites and small reservoirs (17 sites); the DWQ, as part of the statewide ambient water-quality monitoring network, sampled Falls and Jordan Lakes and sites below wastewater-treatment facilities (14 sites). Stream sites were sampled once per month. Lake sites were sampled five times per year. In addition, the USGS sampled each site three times per year for analysis of synthetic organic compounds.

During phase I, several adjustments were made to the network (table 3). The Eno River site at Hillsborough (site 1, Lake Ben Johnson) was moved downstream after the first year

**Table 2.** Water-quality sampling sites in the Triangle Area Water Supply Monitoring Project, North Carolina.

[Sites that were in the original network, beginning in 1988, are shaded; USGS, U.S. Geological Survey; **site type:** I, intake site; W, site below wastewater discharge; T, tributary site; L, in-lake site; Q, discharge only site; WY, water year (defined as the period from October 1 through September 30 and identified by the calendar year in which the period ends); NA, not applicable; cont., continuous through the 2001 water year; DWQ, North Carolina Division of Water Quality; SR, secondary road]

Site number (fig. 2)	USGS station number	Site name and location in North Carolina	County	Drainage area, in square miles	Site type	Routine sampling by	Period of record as of WY2002	
							Streamflow	Water quality (beginning 10/88)
1	0208491605	Eno River at Hillsborough (initial site)	Orange	60.4	I	USGS	NA	10/88–9/89
2	02085000	Eno River at Hillsborough (replaced site 1)	Orange	66.0	I	USGS	10/27–9/71; 10/85–cont.	10/89–cont.
3	02085070	Eno River near Durham	Durham	141.0	W	DWQ	8/63–cont.	10/88–cont.
4	02085079	Eno River near Weaver	Durham	148.0	W	USGS	NA	10/88–9/99
5	0208521324	Little River at SR1461 near Orange Factory	Durham	78.2	T	DWQ	9/61–cont.	10/88–cont.
6	0208524845	Little River Reservoir at dam near Bahama	Durham	97.7	I	USGS	NA	10/88–cont.
7	0208524850	Little River below dam near Fairntosh	Durham	97.8	T	USGS	NA	10/88–6/91
8	02085500	Flat River at Bahama	Durham	149.0	T	DWQ	7/25–cont.	10/88–cont.
9	02086490	Lake Michie at dam near Bahama	Durham	167.0	I	USGS	NA	10/88–cont.
10	02086500	Flat River at dam near Bahama	Durham	168.0	T	DWQ	9/27–cont. <sup>a</sup>	10/88–3/93
11	02086624	Knap of Reeds Creek near Butner	Granville	43.0	W	DWQ	10/82–9/95	10/88–4/95
12	02086849	Ellerbe Creek near Gorman	Durham	21.9	W	DWQ	10/82–9/95	10/88–4/95
13	02086920	Falls Lake at I-85 near Redwood	Durham	512	L	USGS	NA	12/88–7/95
14	0208700780	Little Lick Creek above SR1814 near Oak Grove	Durham	10.1	T	DWQ	10/82–9/95	10/88–4/95
15	0208703650	Falls Lake at N.C. 50 near Sandy Plain	Wake	620	L	USGS	NA	12/88–7/95

**Table 2.** Water-quality sampling sites in the Triangle Area Water Supply Monitoring Project, North Carolina.—Continued

[Sites that were in the original network, beginning in 1988, are shaded; USGS, U.S. Geological Survey; **site type:** I, intake site; W, site below wastewater discharge; T, tributary site; L, in-lake site; Q, discharge only site; WY, water year (defined as the period from October 1 through September 30 and identified by the calendar year in which the period ends); NA, not applicable; cont., continuous through the 2001 water year; DWQ, North Carolina Division of Water Quality; SR, secondary road]

Site number (fig. 2)	USGS station number	Site name and location in North Carolina	County	Drainage area, in square miles	Site type	Routine sampling by	Period of record as of WY2002	
							Streamflow	Water quality (beginning 10/88)
16	0208708905	Falls Lake at N.C. 98 near Bayleaf	Wake	704	L	USGS	NA	12/88–7/95
17	0208718195	Falls Lake above dam at Falls <sup>b</sup>	Wake	771	I	USGS	NA	12/88–7/95
18	02087183	Neuse River near Falls	Wake	771	T	DWQ	6/70–cont.	12/88–9/95
19	02087500	Neuse River near Clayton	Johnston	1,150	Q	NA	8/27–cont.	NA
20	02087570	Neuse River at Smithfield	Johnston	1,206	I	USGS	10/70–9/91	10/88–9/95
21	02087580	Swift Creek near Apex	Wake	21	T	USGS	NA	10/89–7/95
22	02087701	Lake Benson at dam near Garner	Wake	67.0	I	USGS	NA	10/89–6/95
23	02096846	Cane Creek near Orange Grove	Orange	7.5	T	USGS	11/88–cont.	10/88–cont.
24	0209684980	Cane Creek reservoir at dam near White Cross	Orange	31.4	I	USGS	NA	4/89–cont.
25	02096960	Haw River near Bynum	Chatham	1,275	I	DWQ	10/73–cont.	10/88–cont.
26	0209719700	B.E. Jordan Lake, Haw River arm above B.E. Jordan dam	Chatham	1,296	L	USGS	NA	12/88–cont.
27	02097314	New Hope Creek near Blands	Durham	75.9	W	DWQ	10/82–cont.	12/88–cont.
28	0209741955	Northeast Creek at SR1100 near Genlee	Durham	21.1	W	DWQ	10/82–cont.	12/88–cont.
29	02097464	Morgan Creek near White Cross	Orange	8.4	T	USGS	11/82–cont.	10/88–cont.
30	0209749990	University Lake at the dam near Chapel Hill	Orange	30	I	USGS	NA	11/88–cont.

**Table 2.** Water-quality sampling sites in the Triangle Area Water Supply Monitoring Project, North Carolina.—Continued

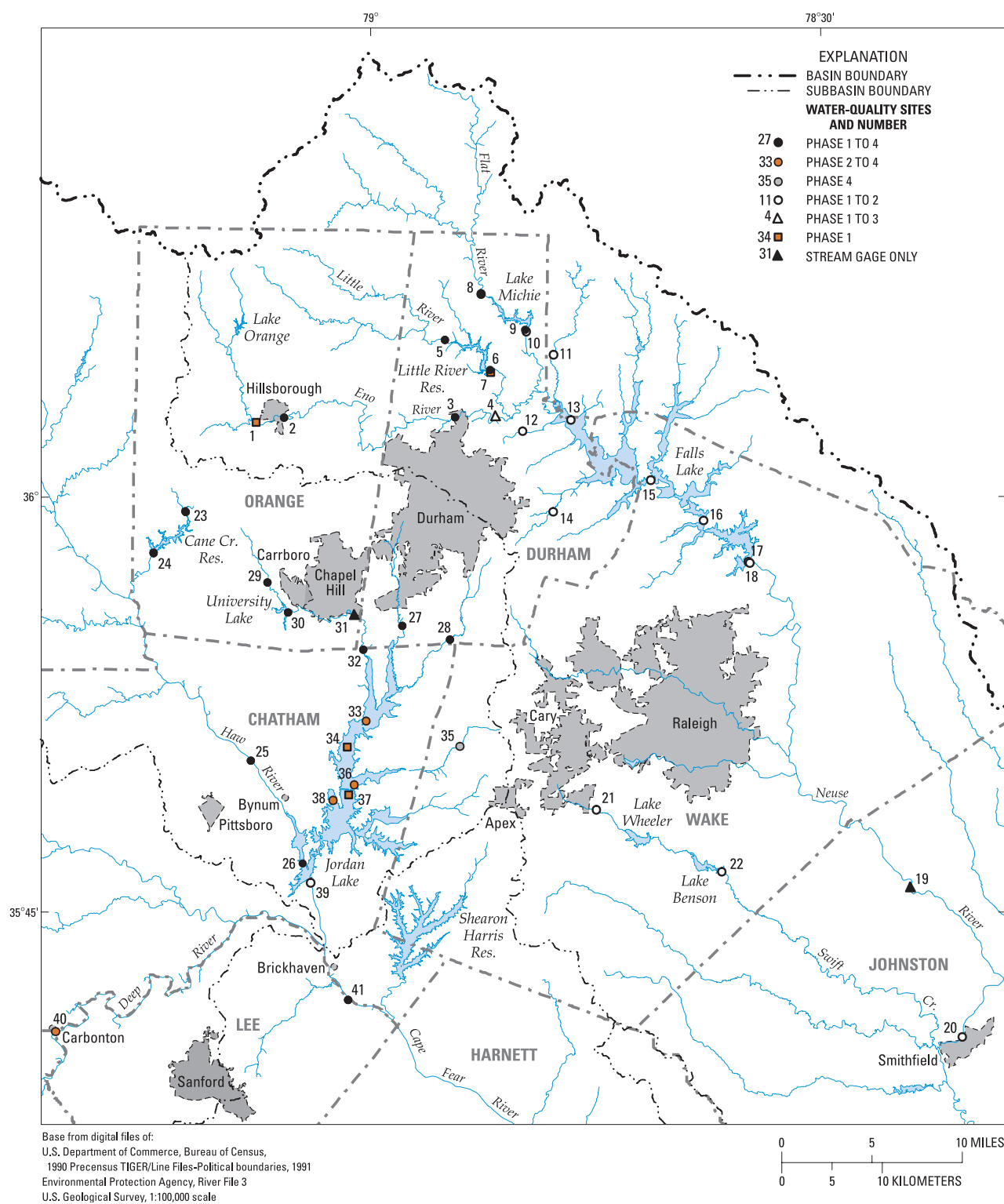
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Site number (fig. 2)	USGS station number	Site name and location in North Carolina	County	Drainage area, in square miles	Site type	Routine sampling by	Period of record as of WY2002	
							Streamflow	Water quality (beginning 10/88)
31	02097517	Morgan Creek near Chapel Hill	Orange	41	Q	NA	11/82–cont.	NA
32	02097521	Morgan Creek near Farrington	Chatham	45.6	W	DWQ	NA	12/88–cont.
33	0209768310	Jordan Lake at buoy 12 at Farrington	Chatham	NA	L	USGS	NA	8/92–cont.
34	0209771550	Jordan Lake at buoy 9 near Farrington	Chatham	250	L	USGS	NA	10/88–9/92
35	0209782520	White Oak Creek at Green Level	Wake	6.97	T	USGS	10/99–cont.	9/99–cont.
36	0209799150	B.E. Jordan Lake above U.S. 64 at Wilsonville	Chatham	NA	I	USGS	NA	7/91–cont.
37	0209801050	Jordan Lake at buoy 7 below U.S. 64 near Griffins Crossroads	Chatham	NA	L	USGS	NA	12/88–9/92
38	0209801100	B.E. Jordan Lake at Bells Landing near Griffins Crossroads	Chatham	NA	I	USGS	NA	7/91–8/95; 8/99–cont.
39	02098198	Haw River below B. Everett Jordan dam near Moncure	Chatham	1,689	T	DWQ	NA	12/88–4/95
40	0210140200	Deep River at Carbondon	Chatham	1,026	I	DWQ	NA	1/93–9/96
41	0210215985	Cape Fear River at N.C. 42 near Brickhaven	Chatham	3,160	I	USGS	NA	10/88–cont.

<sup>a</sup> Includes several multiyear breaks in the record.

<sup>b</sup> Formerly published as USGS station number 02087182.





**Figure 2.** Monitoring sites in the Triangle Area Water Supply Monitoring Project, North Carolina.

# 8 Triangle Area Water Supply Monitoring Project, October 1988 through September 2001, North Carolina

**Table 3.** Water years and phases during which each water-quality site was active in the Triangle Area Water Supply Monitoring network in North Carolina.

[Sites in **bold** are sampled primarily by the U.S. Geological Survey (USGS); SR, secondary road]

Map site no. (fig. 2)	USGS station no.	Site name	Active water years <sup>a</sup> and phases											
			Phase I			Phase II				Phase III				Phase IV
			1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000 2001
1	0208491605	Eno River at Hillsborough (initial site)												
2	02085000	Eno River at Hillsborough (replaced site 1)												
3	02085070	Eno River near Durham												
4	02085079	Eno River near Weaver												
5	0208521324	Little River at SR 1461 near Orange Factory												
6	0208524845	Little River Reservoir at dam near Bahama												
7	0208524850	Little River below dam near Fairintosh												
8	02085500	Flat River at Bahama												
9	02086490	Lake Michie at dam near Bahama												
10	02086500	Flat River at dam near Bahama												
11	02086624	Knap of Reeds Creek near Butner												
12	02086849	Ellerbe Creek near Gorman												
13	02086920	Falls Lake at I-85 near Redwood												
14	0208700780	Little Lick Creek above SR 1814 near Oak Grove												
15	0208703650	Falls Lake at N.C. 50 near Sandy Plain												
16	0208708905	Falls Lake at N.C. 98 near Bayleaf												
17	0208718195	Falls Lake above dam at Falls												
18	02087183	Neuse River near Falls												
20	02087570	Neuse River at Smithfield												
21	02087580	Swift Creek near Apex												
22	02087701	Lake Benson at dam near Garner												
23	02096846	Cane Creek near Orange Grove												
24	0209684980	Cane Creek Reservoir at dam near White Cross												
25	02096960	Haw River near Bynum												
26	0209719700	B.E. Jordan Lake, Haw River arm above B.E. Jordan dam												
27	02097314	New Hope Creek near Blands												
28	0209741955	Northeast Creek at SR 1100 near Genlee												
29	02097464	Morgan Creek near White Cross												
30	0209749990	University Lake at the dam near Chapel Hill												
32	02097521	Morgan Creek near Farrington												
33	0209768310	Jordan Lake at buoy 12 at Farrington												
34	0209771550	Jordan Lake at buoy 9 near Farrington												
35	0209782520	White Oak Creek at Green Level												
36	0209799150	B.E. Jordan Lake above U.S. 64 at Wilsonville												
37	0209801050	Jordan Lake at buoy 7 below U.S. 64 near Griffins Crossroads												
38	0209801100	B.E. Jordan Lake at Bells Landing near Griffins Crossroads												
39	02098198	Haw River below B. Everett Jordan dam near Moncure												
40	0210140200	Deep River at Carbonton												
41	0210215985	Cape Fear River at N.C. 42 near Brickhaven												

<sup>a</sup>Water year is the period from October 1 through September 30 and is defined by the calendar year in which the period ends.

(to site 2). Three more water-supply sites were added to the network in 1991—Lake Benson (site 22, a potential water supply for Raleigh), Jordan Lake (site 36), and Swift Creek near Apex (site 21). In 1991, the DWQ reduced sampling at Falls and Jordan Lakes to once per year, and the USGS began sampling these sites the remaining four times per year as required to meet the project objectives. The unique focus of phase I was to identify the occurrence of synthetic organic compounds in water at the network sites and in Haw River bed sediments.

Phase II of the project began in October 1991 and continued for 4 years through September 1995. Based on the preliminary analysis of the data, the network was adjusted again. The sites downstream from Lake Michie and Little River Reservoir (sites 10 and 7) were discontinued because water quality in the stream channel directly downstream from the lake did not differ substantially from water quality measured in the lakes near the dam. Jordan Lake sites 37 and 34 at buoys 7 and 9, respectively, were discontinued, and site 33 at buoy 12 was added so that the sampling sites in Jordan Lake were distributed among the four segments of Jordan Lake. Also, Deep River at Carbondon (site 40), a water-supply site, was added to the network. In addition to continued routine sample collection, phase II objectives focused on determining if the occurrence of pesticides identified in phase I downstream from municipal wastewater discharges originated from raw wastewater or from sources upstream from the wastewater-discharge point.

Phase III began in October 1995 and continued through June 1999. The City of Raleigh and the Town of Smithfield withdrew funding from the project at the start of phase III. To accommodate the decreased funding, sites located in the Neuse River watershed that were in or downstream from Falls Lake were discontinued as was the Jordan Lake site at Bells Landing (site 38) and Deep River at Carbondon (site 40). In addition, sampling frequency at the remaining sites was decreased from monthly to semimonthly for stream sites and from five times per year to four times per year for lake sites. The special focus of phase III was to assess the occurrence of *Cryptosporidium parvum* oocysts and *Giardia lamblia* cysts in raw-water supplies. Samples were collected from selected sites in the existing network. Sample collection also focused on the collection of water-quality samples during high-flow events.

Phase IV began in July 1999 and continued through June 2003. In this phase, two sites were added—site 35 was added on White Oak Creek at Green Level, a tributary to Jordan Lake, and site 38 was reactivated on Jordan Lake at Bells Landing, the general location of a future water supply. Lake sampling was expanded to include samples for analysis of iron, manganese, and nutrient concentrations at multiple depths. The focus of the phase IV objectives was on increasing the number of high-flow samples that were collected to provide data for use in more accurately estimating constituent load.

Data collected for the Triangle Area Water Supply Monitoring Project have been published annually in USGS data reports for North Carolina (Ragland and others, 1990, 1991; Barker and others, 1992; Gunter and others, 1993; Barker and others, 1994; Ragland and others, 1995–2003). Garrett and

others (1994) published data through 1992. Data collected by the USGS are available to the public in the USGS National Water Information System (NWIS) water-quality database. Interpretations of the data have been reported in unpublished reports by the Triangle Area Water Supply Monitoring Project Steering Committee and in published reports of the USGS (Childress and Treece, 1996; Childress and Bathala, 1997).

## Study Area

The study area is located within a six-county region in the upper Neuse and Cape Fear River basins in the Piedmont Province of central North Carolina (fig. 1). The drainage area of the Neuse River at Smithfield, the downstream terminus of the study area in the Neuse River basin, is 1,206 square miles (mi<sup>2</sup>). The Cape Fear River is formed by the confluence of the Haw (1,695 mi<sup>2</sup>) and Deep (1,441 mi<sup>2</sup>) Rivers downstream from Jordan Lake (fig. 2). The land use in much of the study area is forest or agricultural; however, the cities of Raleigh and Durham and towns of Cary, Chapel Hill, and Hillsborough have significant and growing urban and suburban land covers. The study area is described in more detail in Childress and Treece (1996).

Sampling sites were selected in 1988 to represent the major drinking-water supplies in the area and the major tributaries to the drinking-water supplies (table 2). In 1988, some tributary sites included in the network were being sampled routinely by the North Carolina DWQ as part of the statewide ambient water-quality monitoring network. These sites are identified in table 2. At some of the stream sites, continuous-recording streamflow gages were already established and funded by other agencies. Gages were installed at stream sites with no existing gage.

Twelve water supplies are included in the Triangle Area Water Supply Monitoring Project network (table 4). These raw water supplies are large multipurpose reservoirs (greater than 10,000 acres), small reservoirs (less than 1,000 acres), and run-of-river supplies serving most of the population in the Triangle Area.

The city of Raleigh withdraws drinking water from Falls Lake. Backup water supplies for Raleigh are Lakes Wheeler, Benson, and Johnson. Only Lake Benson was included in this study. Drinking water for the town of Sanford is supplied by the Cape Fear River and for the town of Smithfield by the Neuse River.

The towns of Cary and Apex share a treatment plant that withdraws water from Jordan Lake. Treatment capacity was increased from 24 million gallons per day (Mgal/d) to 40 Mgal/d in 2002 (Town of Cary, 2002). Currently, public surface-water supplies serve less than 10 percent of Chatham County's population. Chatham County purchases treated water from Sanford, Siler City, and Goldston-Gulf Sanitary District and raw water from Cary/Apex. Goldston-Gulf withdraws water from the Deep River.

**Table 4.** Water supplies included in the Triangle Area Water Supply Monitoring Project network in North Carolina.

[OWASA, Orange (County) Water and Sewer Authority]

Raw-water supply	Type of water supply	Municipality or utility	Period in the network through water year 2001
Falls Lake	Large multipurpose reservoir	Raleigh	1988–1995
Jordan Lake at U.S. 64	Large multipurpose reservoir	Cary and Apex	1988–2001
Jordan Lake at Bells Landing	Large multipurpose reservoir	Proposed Chatham County	1991–2001
Lake Michie	Small reservoir	Durham	1988–2001
Little River Reservoir	Small reservoir	Durham	1988–2001
Neuse River at Smithfield	Run of river	Smithfield	1988–1995
Lake Benson	Small reservoir	Raleigh (proposed)	1989–1995
University Lake	Small reservoir	OWASA	1988–2001
Cane Creek Reservoir	Small reservoir	OWASA	1988–2001
Haw River near Bynum	Run of river	Pittsboro	1988–2001
Cape Fear River near Brickhaven	Run of river	Sanford	1988–2001
Deep River at Carbondon	Run of river	Goldston-Gulf Sanitary District	1993–1996
(Lake Ben Johnson) Eno River <sup>a</sup>	Run-of-river reservoir	Hillsborough	1988–2001

<sup>a</sup>Site was moved from the intake to a more suitable sampling location on the Eno River less than 2 miles downstream.

The city of Durham withdraws water from Lake Michie and Little River Reservoir, both in northeastern Durham County. The combined yield of these reservoirs is 43 Mgal/d. The Orange Water and Sewer Authority (OWASA) is a non-profit water and sewer utility that serves the towns of Chapel Hill and Carrboro. OWASA draws its water supply from University Lake and Cane Creek Reservoir. These two lakes can provide up to 15.1 Mgal/d for treatment (Orange Water and Sewer Authority, 2001). Water demand has doubled since 1977 and is expected to double again by 2050. The Quarry Reservoir, west of Carrboro, is available to OWASA for backup use but was not included in this study. Hillsborough is supplied by Lake Orange and Lake Ben Johnson, both impoundments of the Eno River near Hillsborough. Pittsboro is supplied by the Haw River.

Some of the tributaries to the raw-water supplies in the network receive discharges from municipal wastewater-treatment facilities (fig. 1). Among the largest of these are the South Durham Water Reclamation Facility, which discharges to New Hope Creek, and North Durham Water Reclamation Facility (NDWRF), which discharges to Ellerbe Creek. The NDWRF also receives sewage formerly sent to the Eno and Little Lick wastewater-treatment plants, which ceased operation in 1994. Together, these two Durham facilities discharge about 16 Mgal/d. Other wastewater-treatment facilities include the Durham County Triangle Wastewater Treatment Facility, which discharges to Northeast Creek; OWASA's Mason Farms Wastewater Treatment Plant, which discharges about 8 Mgal/d to Morgan Creek; and Raleigh's Neuse River Wastewater Treatment Plant in southeast Raleigh, which discharges about 36 Mgal/d to the Neuse River.

## Sampling and Analytical Methods

Because some project sites were sampled by the USGS and some were sampled by the North Carolina DWQ, methods of data collection and sample analysis differed. The DWQ sample-collection and analysis methods are not described in this report. The USGS sample-collection and analysis methods were in compliance with the USGS standard operating procedures as published in the USGS Techniques for Water-Resources Investigations, in USGS Open-File Reports documenting laboratory methods, and in unpublished USGS North Carolina District quality-assurance guidelines. These publications are referenced in the following sections of this report.

## Field Methods

Field methods encompass sampling activities that take place at the field site, such as sample collection, processing and preservation of samples, and in-situ or onsite measurement of physical properties. USGS field protocols are contained in various published and unpublished USGS documents and memorandums (Guy and Norman, 1970; Office of Water Data Coordination, 1977; U.S. Geological Survey, 1997–present; Edwards and Glysson, 1999; North Carolina District Quality-Assurance Plan for Water Quality, written commun., 2000). The North Carolina District quality-assurance plan for water-quality activities (North Carolina District Quality-Assurance Plan for Water Quality, written commun., 2000) and quality-assurance plan for surface-water activities (North Carolina District Quality-Assurance Plan for Surface Water, written commun., 1995) are updated approximately every 3 years. These plans

provide guidance to USGS staff on water-quality and surface-water field methods and include references to relevant USGS memorandums.

## Sample Collection

At each stream site, dissolved oxygen and temperature were measured in situ. Specific conductance and pH were measured in situ or in the composited sample on site. Measurements were made according to USGS published and unpublished protocols (U.S. Geological Survey, 1997–present; North Carolina District Quality-Assurance Plan for Water Quality, written commun., 2000).

Samples were collected and processed according to standard USGS protocols in effect at the time of sample collection. A major revision in USGS protocols for collecting samples occurred in 1994 when new sample-handling techniques with less potential for sample contamination were introduced. The so called "parts-per-billion protocol" or "clean-hands protocol" was developed for the collection of samples containing low-level concentrations of trace elements and nutrients. These methods were published in the USGS National Field Manual (U.S. Geological Survey, 1997–present) and were adopted for use in this study.

## Streams

At each stream site, measurements of dissolved oxygen, temperature, specific conductance, and pH were measured in situ or in the collected sample (except dissolved oxygen). Measurements were made electrometrically with individual meters or with a multiparameter data sonde. Alkalinity was measured at the time of sample collection using fixed-endpoint (to pH 4.5) or incremental titration.

Stream samples usually were collected with depth-integrating isokinetic samplers (fig. 3) using equal-width-increment (EWI) methods (Edwards and Glysson, 1999; Wilde and others, 1999a). The EWI method provides for collection of a sample that is representative of the average concentration in the stream cross section. Isokinetic samplers were designed by the Federal Interagency Sedimentation Project (FISP) to allow flowing water to enter the sampler nozzle at the same velocity as in the stream. Improved designs for water-quality samplers were introduced during the course of the project and were adopted as they became available.

For wadeable streams, the samplers that were used included the 22-pound (lb), plastic-coated, bronze DH-59 sampler with a 1-pint glass bottle that was used prior to about 1990, and the 0.5-lb polypropylene DH-81 sampler with a 1-liter (L) polyethylene bottle (fig. 3). For wadeable streams, suspended-sediment samples were collected separately from the water-quality samples using an aluminum DH-48 (not pictured) or the DH-59 sampler with a pint-size glass bottle.

For streams too deep to wade, sampling was done from a bridge using a 22-lb, plastic-coated, bronze DH-76 sampler with a 1-quart glass bottle; a 75-lb, plastic-coated, bronze D-77

cable-suspended sampler with 1- or 3-L polyethylene bottle; or a 64-lb, plastic-coated, bronze D-95 sampler with a 1-L polyethylene bottle. Use of the DH-76 sampler for collection of water-quality samples was discontinued in about 1992. Use of the D-77 sampler for collection of water-quality samples was discontinued in about 1998. Suspended-sediment samples were collected separately from the water-quality samples by using a DH-76 or DH-59 sampler with a quart-size and pint-size glass bottle, respectively. All of the isokinetic samplers are fitted with nylon or Teflon nozzles. The DH-81, D-77, and D-95 samplers are designed so that the nozzle fits into a plastic or Teflon cap that threads directly onto the sample bottle, which minimizes contact between the sampler and the sampled water. Details about sampler design are provided in Wilde and others (1998b) and Edwards and Glysson (1999).

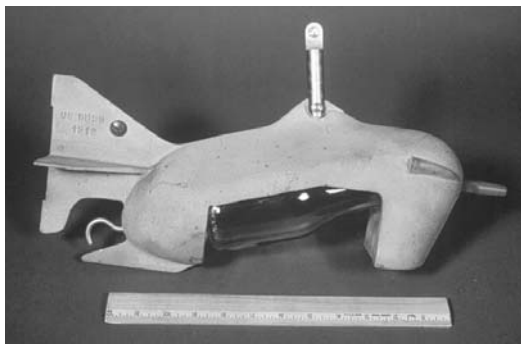
Isokinetic samplers are effective when stream velocity exceeds 1.5 feet per second (ft/s) and stream depth exceeds about 0.5 foot (ft; Wilde and others, 1998b). When stream velocity was less than 1.5 ft/s or depth was less than 0.5 ft, multiple subsamples were collected at equal widths in the stream cross section using a weighted bottle sampler (also called a sewage sampler) or bottles were filled directly by dipping them in the stream (dip method).

As multiple subsamples were collected as described above using isokinetic or weighted bottle samplers or the dip method, subsamples were composited in an 8-L plastic churn splitter before 1994 and in a modified 8-L plastic churn splitter after 1994 (Wilde and others, 1998b, Section 2.2.1A). The modifications to the churn splitter were designed to minimize the potential for atmospheric contamination to the sample and to improve the spigot mechanism to reduce the potential for contamination when the sample was withdrawn from the churn into the sample bottle.

Suspended-sediment samples were collected only at stream sites. Samples were collected by using the EWI methods described in Guy and Norman (1970) and Edwards and Glysson (1999) at multiple locations in the stream cross section. Suspended-sediment subsamples were not composited onsite but were sent instead to the sediment laboratory in the individual bottles in which they were collected. Sample bottles were shipped to the sediment laboratory in crates without preservation.

Samples for analysis of total organic carbon and synthetic organic compounds could not be collected with the isokinetic samplers described above and could not be composited in a churn splitter because of potential contamination from plastic components in the samplers, bottles, and churn splitter. Instead, samples for analysis of total organic carbon and synthetic organic compounds were collected directly into baked-glass sample bottles using a weighted bottle sampler or the dip method of sample collection. These samples were collected at the center of flow in the stream.

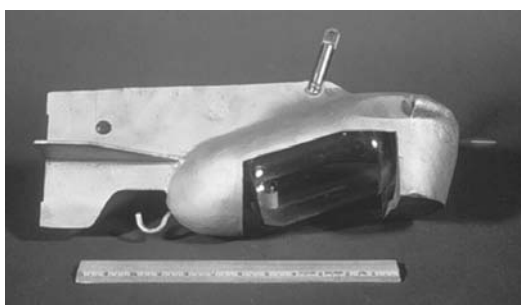
Hand samplers



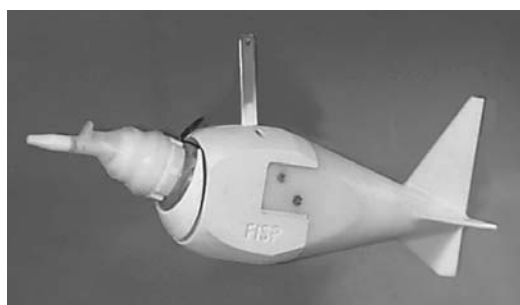
DH-59



DH-81

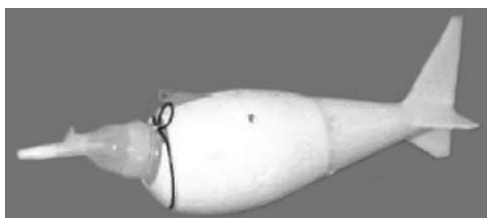


DH-76

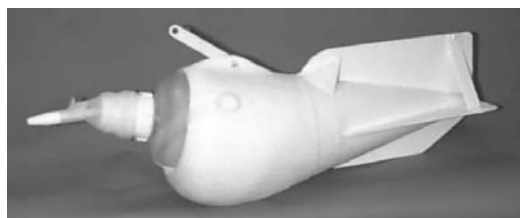


DH-95

Cable suspended samplers



D-95



D-77

**Figure 3.** Selected water-quality and suspended-sediment samplers used in streams and rivers for the Triangle Area Water Supply Monitoring Project, North Carolina.

## Lakes

At each lake site, measurements of dissolved oxygen, temperature, specific conductance, and pH were measured at 1-meter (about 3.3-ft) vertical intervals from the surface to the lake bed. A multiparameter data sonde was used to measure and record simultaneous readings of each constituent.

Lake samples were collected by using the dip sample method, a Van Dorn horizontal thief sampler (Wilde and others, 1998b), or a Lab-line™ sampler (fig. 4). The Lab-line™ sampler consists of a vertical cylinder that opens only at the top. Two small ports in the top are for water intake and for air exhaust. Caps on each opening can be removed at a specific depth or the ports can be left uncapped so that the chamber fills as the sampler is lowered vertically through the water column.

Prior to 1992, all lake samples were collected at about 1 meter (about 3.3 ft) below the water surface using the Van Dorn sampler. Near the beginning of phase II, in April 1992, lake-sampling methods used by the USGS were changed to match methods used by the DWQ for lake sampling. That is, nutrient and chlorophyll *a* samples were collected with the Lab-line™ sampler leaving the two ports uncapped in order to collect a sample integrated throughout the water column to a depth of twice the secchi depth reading. Other organic and inorganic constituents were collected by using the dip method

at about 0.3 meter (about 0.98 ft) below the surface. Beginning in phase IV, additional samples for nutrients, chlorophyll *a*, iron, and manganese were collected using the Van Dorn sampler at the thermocline and at about 1 meter (about 3.3 ft) from the lake

bottom. The location of the thermocline was determined from measurements of dissolved-oxygen concentrations taken at 1-meter (about 3.3-ft) intervals throughout the water column.

Prior to October 1989, samples from University Lake and the Eno River at Hillsborough were collected directly from a tap on the municipal water-supply intake line. This resulted in copper contamination because the intake line was made of copper. In October 1989, both sampling locations were moved so that the samples were collected directly from the raw-water supply.

## Sample Processing

Each piece of sampling equipment that had contact with sample water (churn splitters, sample-collection bottles, sampler nozzles, peristaltic pump hose, Lab-line™ or Van Dorn samplers) was cleaned with a laboratory-grade phosphate-free detergent, rinsed with tap water, and given several final rinses with de-ionized water produced in the USGS North Carolina District office. The parts-per-billion protocol introduced in 1994 required more rigorous cleaning, including an additional soak in a 5-percent hydrochloric acid solution prior to the final de-ionized water rinse, and careful packaging in a plastic bag after cleaning to maintain cleanliness (U.S. Geological Survey, 1997).

Sample processing includes compositing EWI subsamples, filtering the sample through a 0.45-micron ( $\mu$ ) membrane filter, filling sample bottles, and preserving the samples. All sample processing for stream and small lake sites was completed onsite and in accordance with USGS protocols and requirements specified by the USGS National Water Quality Laboratory (NWQL) for the particular analyses being requested (table 5). Samples collected from a boat on Falls and Jordan Lakes were processed either onboard the boat onsite or in the field vehicle after completing all lake-sample collections. The decision on where to process these samples depended on lake conditions at the time of sampling.

Lab-line sampler



Van Dorn sampler



**Figure 4.** Lake samplers used for the Triangle Area Water Supply Monitoring Project in North Carolina.

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**Table 5.** Sample schedules, bottle requirements, and preservation of samples analyzed at the U.S. Geological Survey National Water Quality Laboratory.

[NWQL, National Water Quality Laboratory; mL, milliliter; poly., polyethylene; raw, unfiltered sample; chilled, held on ice to a temperature of 4 degrees Celsius (°C); HgCl, mercuric chloride tablet added; filtered, poured through a 0.45–micron pore size membrane filter; HCl, hydrochloric acid; 4.5N H<sub>2</sub>SO<sub>4</sub>, 4.5 normal sulfuric acid was added to pH less than (<) 2; HNO<sub>3</sub>, nitric acid added to pH < 2; 6N HCl, 6 normal ultra-pure hydrochloric acid was added to pH < 2; KCr<sub>2</sub>, potassium dichromate preservative was added; L, liter; baked, heated in oven to 450 °C; µg/L, microgram per liter]

Schedule description and notes	NWQL schedule number and(or) lab code (LC)	Period of use (in water year)	Bottles required	Treatment and preservation
<b>Nutrients</b> Nutrients consist of nitrite plus nitrate, nitrite, organic nitrogen plus ammonia, ammonia, orthophosphorus, and total phosphorus 1989 – 1993 samples treated with HgCl 1994 – 1999 samples chilled only 1999 – total Kjeldahl nitrogen and phosphorus sample treated with HCl 1994 – lab discontinued analysis of unfiltered nitrite, nitrite plus nitrate, ammonia, and orthophosphate	5	10/88 – 9/91	125–mL brown, poly.	Raw, chilled, HgCl.
			125–mL brown, poly.	Filtered, chilled, HgCl.
	535	10/91 – 9/96	125–mL brown, poly.	Raw, chilled, HgCl (until 9/94); raw, chilled (beginning 10/94).
			125–mL brown, poly.	Filtered, chilled, HgCl (until 9/94); filtered, chilled (beginning 10/94).
	943 plus LC1984	10/96 – 9/01	125–mL brown, poly. (until 12/98); 125–mL clear poly. (beginning 1/99)	Raw, chilled (beginning 10/94); raw, 1 mL 4.5N H <sub>2</sub> SO <sub>4</sub> , chilled (beginning 1/99).
			125–mL brown, poly.	Filtered, chilled.
<b>Major ions and other standard constituents</b> Major ions consist of calcium, sodium, potassium, magnesium, chloride, sulfate, bicarbonate fluoride, and silica. Also includes color, residue at 180 °C, pH, specific conductance, and total organic carbon	1030	10/88 – 9/91	250–mL poly., acid rinsed	Raw, untreated.
			250–mL poly., acid rinsed	Filtered, HNO <sub>3</sub> to pH < 2.
			250–mL poly.	Raw, chilled.
	823	10/91 – 9/01	500–mL poly., acid rinsed	Raw, HNO <sub>3</sub>
			250–mL poly., acid rinsed	Raw, HNO <sub>3</sub>
			250–mL glass, acid rinsed	Raw, 6N HCl.
<b>Trace metals</b> Aluminum, arsenic, cadmium, chromium, copper, iron, lead, manganese, mercury, molybdenum, nickel, selenium, silver, and zinc	819	10/88 – 9/01	250–mL, poly., acid rinsed	Raw, HNO <sub>3</sub> to pH < 2.
			250–mL, glass, acid rinsed	Raw, KCr <sub>2</sub> (until 4/01) Raw, 2 mL 6N HCl.
Phytoplankton chlorophyll by high-performance liquid chromatography (HPLC)	1508	10/88 – 9/01	Amber glass vial	Known volume filtered with glass-fiber filter; stored in bottle wrapped in foil, chilled.
Insecticides: organochlorine with gross polychlorinated biphenyls (PCBs) and gross polychlorinated naphthalenes (PCNs), and organophosphates, by gas chromatography (GC) electron capture detector (ECD) and GC flame photometric detector (FPD)	1334	10/88 – 1993	1–L amber baked glass	Chilled.



**Table 5.** Sample schedules, bottle requirements, and preservation of samples analyzed at the U.S. Geological Survey National Water Quality Laboratory.—Continued

[NWQL, National Water Quality Laboratory; mL, milliliter; poly., polyethylene; raw, unfiltered sample; chilled, held on ice to a temperature of 4 degrees Celsius (°C); HgCl<sub>2</sub>, mercuric chloride tablet added; filtered, poured through a 0.45-micron pore size membrane filter; HCl, hydrochloric acid; 4.5N H<sub>2</sub>SO<sub>4</sub>, 4.5 normal sulfuric acid was added to pH less than (<) 2; HNO<sub>3</sub>, nitric acid added to pH < 2; 6N HCl, 6 normal ultra-pure hydrochloric acid was added to pH < 2; KCr<sub>2</sub>O<sub>7</sub>, potassium dichromate preservative was added; L, liter; baked, heated in oven to 450 °C; µg/L, microgram per liter]

Schedule description and notes	NWQL schedule number and(or) lab code (LC)	Period of use (in water year)	Bottles required	Treatment and preservation
Semivolatiles, total, acid, base, and neutral extractable compounds by GC mass spectrometry (MS)	1383	10/88 – 1992	1–L amber baked glass	Chilled.
Organic compounds scanned by GC flame ionization detector (FID)	1381	10/88 – 1993	1–L amber baked glass	Chilled.
Volatile organic compounds at 0.2 µg/L by purge and trap—GC/MS	1391	10/88 – 9/91	3 septum vials of 40 mL each	Chilled.
Low-level organophosphate and organochlorine pesticides and gross PCBs	1399	1989 – 1991	1–L amber baked glass	Chilled.
Triazine herbicides in filtered water by GC/MS	1379	1992 – 1993	1–L amber baked glass	Chilled.
Pesticides in filtered water (Carbopak-B solid-phase extraction cartridge - Lab), HPLC	2050	1996, 1998	1–L amber baked glass	Chilled.
Pesticides in filtered water (C-18 solid-phase extraction cartridge - Lab), GC/MS	2001	1996, 1998	1–L amber baked glass	Chilled.
Volatile organic compounds by purge and trap—GC/MS	2091	1996, 1998	3 septum vials of 40 mL each	Bottles filled, with no air bubbles, chilled.

First, sample bottles for constituents that do not require filtration, such as total metals, total nutrients, and major ions, were filled by using the paddle of the churn splitter to mix the composited sample while a subsample was withdrawn into the sample bottle. Next, sample bottles for constituents that require filtration were filled. A peristaltic pump was used to pump the composite sample from the churn splitter through a filter and into the sample bottle. An inert tubing, usually C-Flex™, was used to pump water through a 142-millimeter (mm) 0.45-µ, cellulose-nitrate membrane filter using a peristaltic pump and Geotech® polycarbonate filter holder (before 1994) or through a Gelman® 0.45-µ, polysulfonate, pleated-membrane, disposable, polypropylene capsule filter (after 1994). The filter was pre-rinsed with de-ionized water before use.

Samples for analysis of chlorophyll *a* and *b* concentrations were processed by filtering a known volume of sample (typically 100 milliliters [mL]) through a 0.45-µ, glass-fiber filter. A pair of forceps was used to remove the filter from the filter screen. The filter then was folded, placed in a plastic petri dish or amber glass vial, wrapped in aluminum foil to exclude light (petri dish only), and placed on ice for shipping. The volume of water that was filtered was recorded on the petri dish or vial.

Samples for the analysis of synthetic organic compounds were sent to the laboratory unfiltered. Filtering of these samples, when required, was done at the laboratory. Samples were collected in brown, baked-glass bottles and placed on ice for shipping.

## Streamflow Measurement

Instantaneous streamflow at the time of sample collection was obtained by applying a site-specific stage-discharge relation to the stage record at stream sites in the network that were equipped with a continuous record gage (table 2). Rantz and others (1983) describe USGS methods for operating and maintaining streamflow-gaging stations by developing a stage-streamflow relation. Streamflow record is developed from periodic instantaneous measurements of stage and streamflow that are used to construct a stage-streamflow relation. The stage-streamflow relation is used to calculate continuous streamflow from 15- to 60-minute-interval stage record.

Streamflow for Morgan Creek near Farrington (site 32, fig. 2) was estimated from streamflow measured by the upstream gage at Chapel Hill (about a 10-percent difference in

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drainage area). Streamflow for the Neuse River at Smithfield (site 20, fig. 2) was estimated from the Neuse River gage at Clayton (about a 5-percent difference in drainage area). Streamflow for the Cape Fear River near Brickhaven (site 41, fig. 2) was estimated by adding daily mean releases from Jordan Lake reported to the USGS by the U.S. Army Corps of Engineers and the mean daily streamflow from the gage on the Deep River at Moncure. Beginning in 1992, instantaneous streamflow was measured at the time of sample collection at Swift Creek at Apex and the Neuse River at Smithfield. To measure streamflow, a Pygmy or Price AA velocity meter was used (Rantz and others, 1983). Most sampling sites without gages were discontinued by 1995.

A goal of the project is to sample water quality for a range of streamflow conditions at each site with particular attention to sampling during high-flow events when most of the constituent load of concern is transported. Over the long term, the fixed-interval monitoring design of this project (once per month or once per 2 months) was expected to produce the desired range of conditions. However, to ensure that sufficient high-streamflow events are sampled for adequate definition of water-quality conditions during high streamflow, an additional five samples per year specifically were targeted for high-streamflow events.

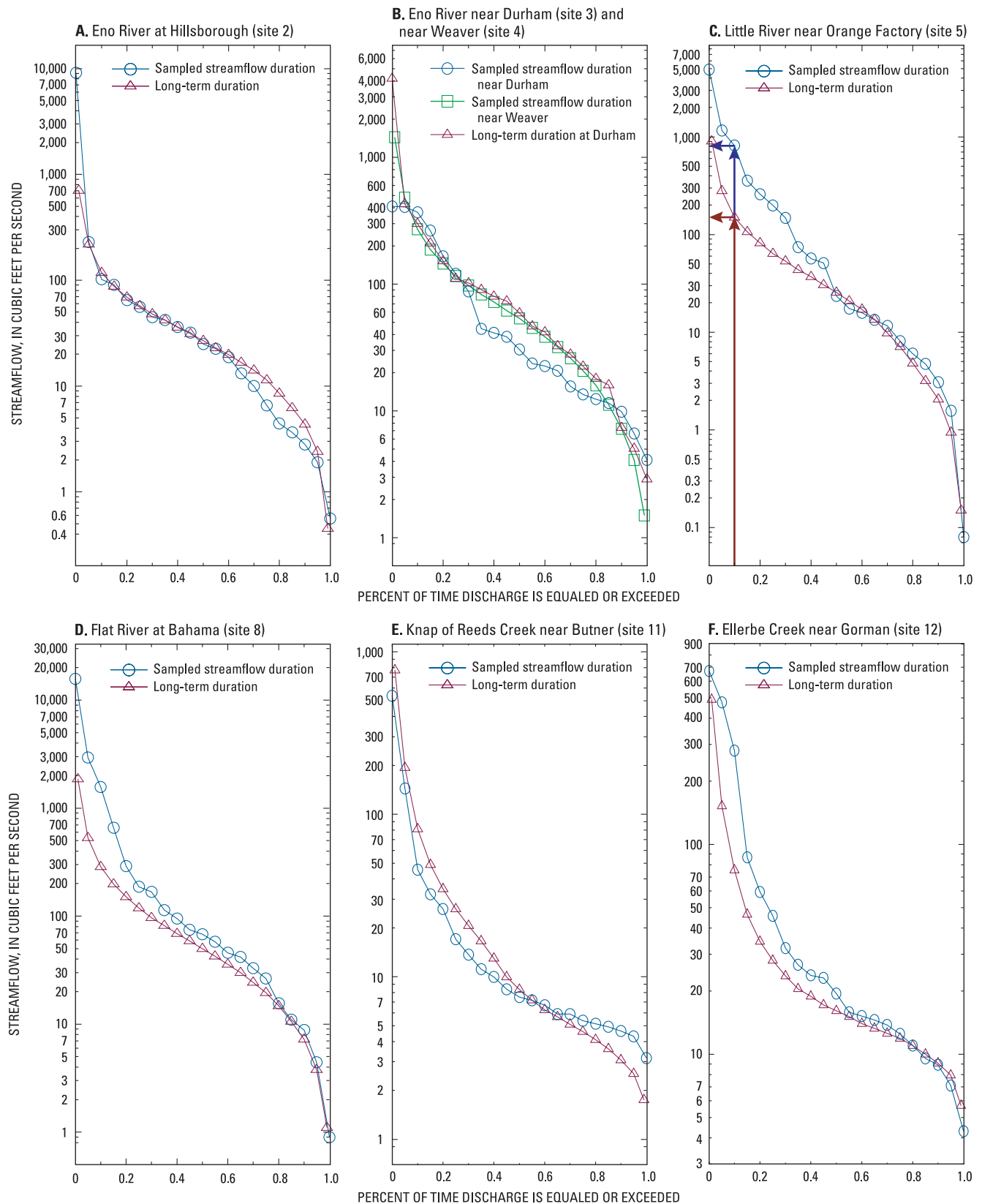
Streamflow durations were used to assess whether high streamflows were sampled during the study at a frequency

comparable to or greater than the long-term frequency of high-streamflow events. A streamflow-duration curve indicates the percentage of time that specific streamflow quantities are equaled or exceeded based on a given period of record. Figure 5 illustrates a comparison of long-term streamflow durations—streamflow durations based on the entire period of record at a gage—with streamflow durations based only on streamflows sampled during the study. Using Little River near Orange Factory (site 5, fig. 2) as an example, a streamflow of 150 cubic feet per second ( $\text{ft}^3/\text{s}$ ) was equaled or exceeded about 10 percent of the time based on long-term record (fig. 5C). By comparison, a streamflow of about 800  $\text{ft}^3/\text{s}$  was equaled or exceeded about 10 percent of the time based on streamflow sampled for the study. Therefore, the streamflows that were sampled were somewhat biased toward high-streamflow events.

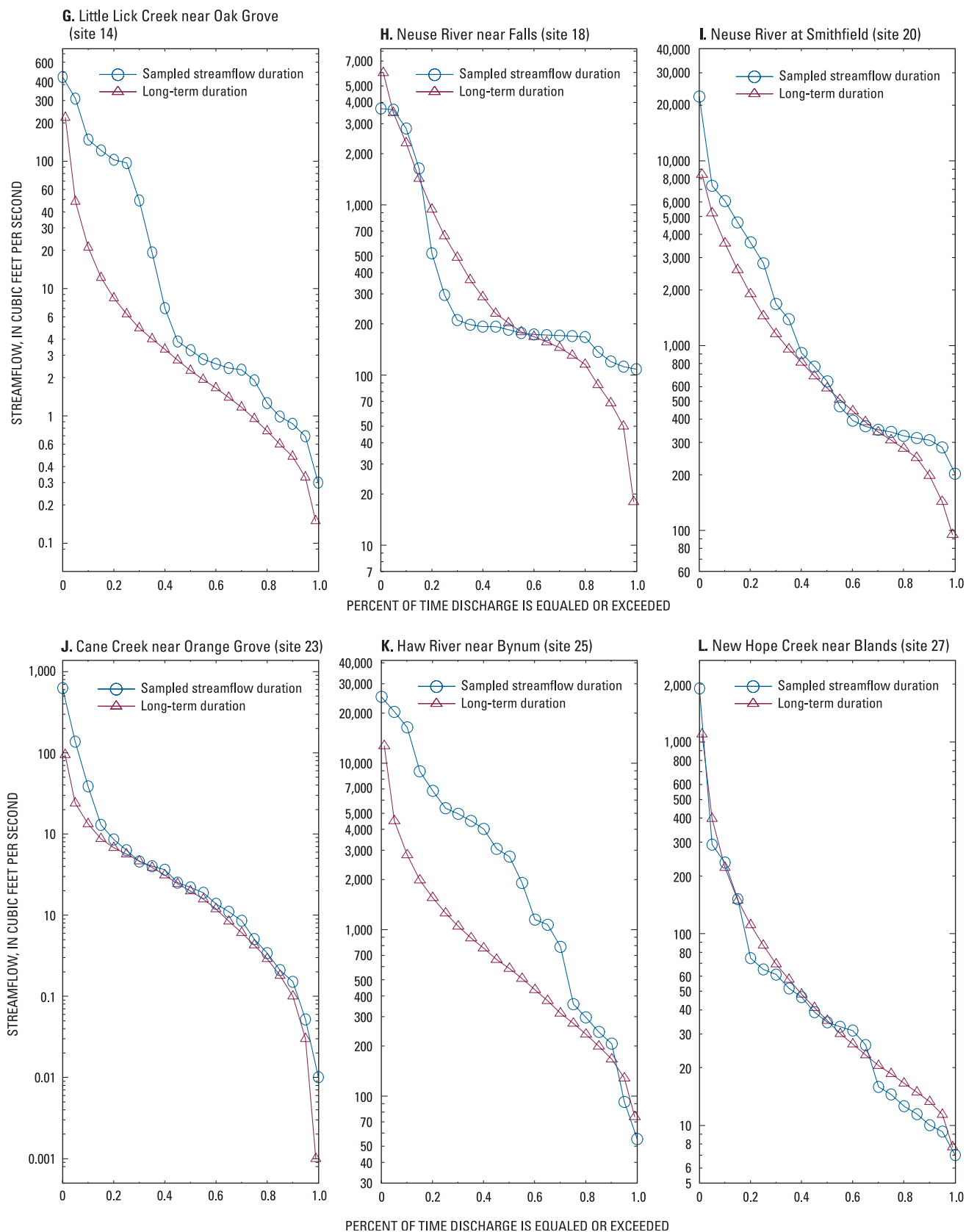
In general, when the duration curve for sampled streamflows is above the long-term duration curve, as it was for Little River near Orange Factory, sampling was somewhat biased toward high-streamflow events. When the sampled duration curve is below the long-term duration curve, as it was for Morgan Creek near Farrington (site 32, fig. 5O) and Eno River near Durham (site 3, fig. 5B), sampling was somewhat biased toward low-streamflow periods. The goal was to bias sampling slightly toward high-streamflow events because most transport of sediments, nutrients, and other constituents associated with sediment occurs during high-flow events.



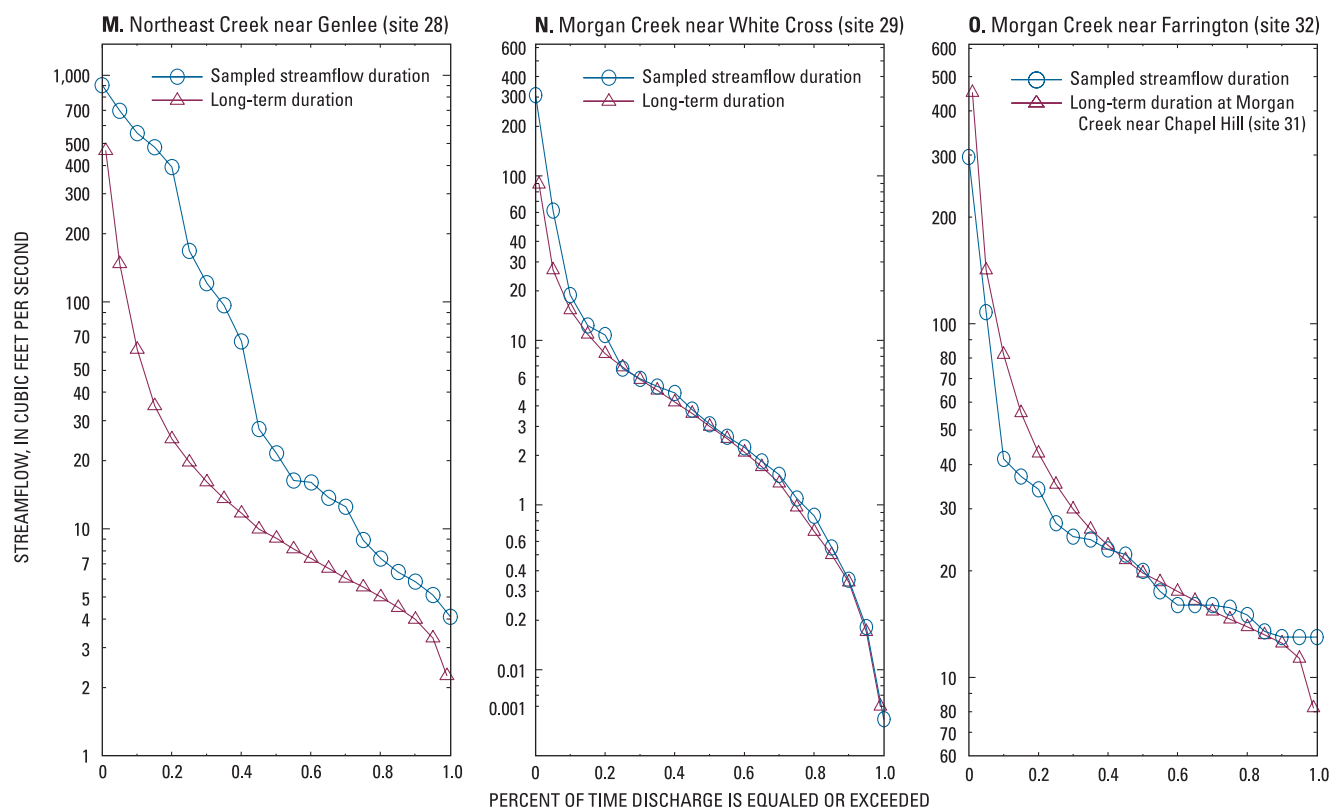
A USGS hydrologic technician collecting a water-quality sample.



**Figure 5.** Comparison of streamflow durations from long-term streamflow record with durations for sampled streamflow conditions.



**Figure 5. (Continued)** Comparison of streamflow durations from long-term streamflow record with durations for sampled streamflow conditions.



**Figure 5. (Continued)** Comparison of streamflow durations from long-term streamflow record with durations for sampled streamflow conditions.

## Laboratory Analysis

The DWQ Laboratory Section analyzed samples collected by the DWQ. The constituents generally analyzed in water samples collected by the DWQ at the ambient water-quality monitoring network sites and the frequency of sample collection are listed in table 6. The NWQL in Lakewood, Colorado, analyzed all water-quality samples collected by the USGS (tables 7, 8, p. 45–56) except for suspended-sediment samples. Samples were processed and preserved onsite according to methods used by the NWQL (table 5). Sample bottles were packed on ice and shipped to the laboratory using overnight express-delivery service. Once received at the laboratory, samples were logged into the laboratory computer system and assigned a laboratory identification number.

Suspended-sediment samples were analyzed by the USGS North Carolina District sediment laboratory prior to 1996 and by the USGS Kentucky District sediment laboratory thereafter. Samples sent to Kentucky for analysis were boxed and shipped to the laboratory without preservation. Samples were analyzed by the method described in Guy (1969).

## Quality-Assurance and Quality-Control Practices

Quality assurance and quality control encompass the activities designed to define the quality of the data collected for the project. Quality assurance associated with this project included adherence to standard USGS protocols for sample collection and processing, periodic review of field activities by the senior water-quality technician or water-quality specialist in the USGS North Carolina District, and participation by field staff in the USGS Field Quality-Assurance Program. This program provides field staff with once- or twice-yearly unknown samples for analysis of pH, specific conductance, and alkalinity. In addition, USGS project technical review includes reviews of project activities by District management and senior technical staff at least three times per year and triennial reviews conducted by the USGS Water-Resources Discipline, Office of Water-Quality.

Quality-control activities included the collection of blank and replicate samples for chemical analysis, use of standard

**Table 6.** North Carolina Division of Water Quality sampling schedule and analytical methods for sites in the Triangle Area Water Supply Monitoring Project.

[QW, water quality; °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25 °C; std units, standard units; mg/L, milligram per liter; N, nitrogen; P, phosphorus; AA, atomic absorption spectrometry; µg/L, microgram per liter]

Constituent	Reporting units	Sampling frequency (samples per year)	Analytical method	Detection limit
<b>Physical properties and general QW</b>				
Temperature	°C	10 – 12	Thermometer/thermistor	0.5
Specific conductance	µS/cm	10 – 12	Electrometric	1
pH	std units	10 – 12	Electrometric	0.1
Dissolved oxygen	mg/L	10 – 12	Electrometric	0.1
Hardness	mg/L	3 – 4	titration	1
Residue on evaporation at 105 °C, suspended	mg/L	3 – 4	Gravimetric	1
Residue on evaporation at 105 °C, total	mg/L	3 – 4	Gravimetric	1
Chlorophyll <i>a</i>	mg/L	3 – 4	Fluorometric	1
Chlorophyll <i>b</i>	mg/L	3 – 4	Fluorometric	1
<b>Nutrients</b>				
Total nitrogen as N	mg/L	10 – 12	Calculated	
Total ammonia plus organic nitrogen as N	mg/L	10 – 12	Automated phenate, colorimetry	0.1
Total ammonia as N	mg/L	10 – 12	Automated phenate, colorimetry	0.01
Total nitrite plus nitrate as N	mg/L	10 – 12	Cadmium reduction, automated, colorimetry	0.01
Total phosphorus as P	mg/L	10 – 12	Persulfate digestion, automated, ascorbic acid reduction, colorimetry	0.01
Total orthophosphorus as P	mg/L	10 – 12	Ascorbic acid reduction, automated, colorimetry	0.01
<b>Trace elements</b>				
Arsenic	mg/L	3 – 4	Digestion, AA, graphite furnace	10
Cadmium	µg/L	3 – 4	Digestion, AA, graphite furnace	2
Chromium	µg/L	3 – 4	Digestion, AA, direct aspiration	25
Copper	µg/L	3 – 4	Digestion, AA, direct aspiration	10
Lead	µg/L	3 – 4	Digestion, AA, graphite furnace	10
Mercury	µg/L	3 – 4	Digestion, AA, cold vapor	0.2
Nickel	µg/L	3 – 4	Digestion, AA, graphite furnace	10
Zinc	µg/L	3 – 4	Digestion, AA, direct aspiration	10

protocols for cleaning and storing equipment and supplies, and the purchase and use of preservatives and reagents that are quality assured by the USGS Field Services Unit in Ocala, Florida.

## Supplies

The supplies needed for processing samples generally were purchased from USGS sources. Prior to 1993, most supplies were purchased from the NWQL. The USGS Field Services Unit, in Ocala, Florida, assumed most of the supply service responsibilities from the NWQL beginning in 1993. In both cases, supplies were purchased in bulk, and each lot was quality checked before distribution to USGS offices. Such supplies included sample preservatives, alkalinity reagents, inorganic blank reagent water, organic blank water (from the NWQL only), capsule filters, and sample bottles. Each lot of chemical reagents, bottles, and filters was tested for contamination and performance quality prior to distribution. Bottles were acid rinsed or baked, as required.

De-ionized water produced in the USGS North Carolina District laboratory was used for rinsing equipment and supplies. To produce de-ionized water, a purifying system was installed in which tap water was passed through a 1- $\mu$  pre-filter, two de-ionizing columns placed in series, and a 0.2- $\mu$  post filter. The specific conductivity of the water after passage through the first de-ionizing column was monitored continually with an indicator light to determine when resistance was less than 17 megaohms per centimeter (greater than 0.06 microsiemens per centimeter ( $\mu$ S/cm) at 25 degrees Celsius). The first de-ionizing column in the series was replaced every 6 months or when the indicator light indicated that specific conductance had exceeded the criterion. The second de-ionizing column in the series replaced the first de-ionizing column, and a new de-ionizing column was added to the second position. To produce field blanks for inorganic constituents, de-ionized water, certified as free from inorganic contaminants, was purchased from the NWQL or the Ocala Field Services Unit.

## Record Management and Archive

Water-quality records were managed, stored, and archived according to the USGS North Carolina District quality-assurance guidelines for water-quality data (North Carolina District Quality-Assurance Plan for Water Quality, written commun., 2000). Streamflow records were managed, stored, and archived according to the USGS North Carolina District quality-assurance guidelines for surface-water data (North Carolina District Quality-Assurance Plan for Surface Water, written commun., 1995). All water-quality and streamflow records are stored in the USGS National Water Information System (NWIS) and are available to the public at <http://waterdata.usgs.gov/nwis/>.

## Field Notes

Field notes consist of written observations of field conditions, field instrument-calibration notes, water-quality field measurements, documentation of sample preservation and processing steps, and record of samples collected for shipment to the laboratory. Field notes are filed in folders according to station number and water year and stored at the USGS North Carolina District office. Record of sample-collection date, time, and field conditions and record of field measurements are entered by field staff into the NWIS database.

## Water-Quality Analytical Results

Water-quality analytical results are transferred electronically to the USGS North Carolina District NWIS database from the NWQL. Paper copies of analytical results are printed and filed with the field notes in folders according to station number and water year. Suspended-sediment concentration results are reported from the laboratory in a written report. Results are entered in the NWIS database, and the written report is filed with other sample records for the same sample event.

Analytical results are reviewed for consistency by the NWQL quality-assurance unit and, upon receipt, by the USGS North Carolina District water-quality specialist and project chief. If results are judged to be questionable, the laboratory is asked to verify the results and, if necessary, rerun the sample analysis. When this occurs, the new result is compared with the original result, and the result judged most reasonably correct is entered into the database.

Gas chromatography with flame ionization detector (GC/FID) scans for synthetic organic analysis were qualitative, and only a paper copy of the results of the scan was provided by the laboratory. The qualitative results—copies of gas chromatography traces and results of a library search for probable identification of peaks on the chromatograph—were filed and archived with the other project paper records.

## Streamflow Records

Streamflow-gaging stations were operated and maintained according to established USGS protocols (Rantz and others, 1983) and North Carolina District guidelines (North Carolina District Quality-Assurance Plan for Surface Water, written commun., 1995). Records of daily mean streamflow are stored permanently in the NWIS database. Records of unit values (15- to 60-minute intervals) of stage and streamflow are stored permanently in the NWIS database with the exception of a few years when electronically stored data were lost because of a computer error (table 9). Paper copies of unit-value discharge data for these periods are available in the USGS North Carolina District office.

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**Table 9.** Status of electronic storage of unit-value data for water years 1989–2001 in the U.S. Geological Survey National Water Information System database for the Triangle Area Water Supply Monitoring Project in North Carolina.

[USGS site number, the downstream order number assigned by the U.S. Geological Survey to each streamflow-gaging site; U, unit-value data stored electronically; X, unit-value data available only on paper copies; SR, secondary road; shaded area indicates years with no streamflow gage]

USGS site number	USGS site name	Water year												
		89	90	91	92	93	94	95	96	97	98	99	00	01
02085000	Eno River at Hillsborough	U	U	U	U	X	U	U	U	U	U	U	U	U
02085070	Eno River near Durham	U	U	U	X	X	U	U	U	U	U	U	U	U
0208521324	Little River at SR 1461 near Orange Factory	U	U	U	U	X	U	U	U	U	U	U	U	U
02085500	Flat River at Bahama	U	U	U	X	X	U	U	U	U	U	U	U	U
02086624	Knap of Reeds Creek near Butner	U	U	U	U	X	U	X						
0208700780	Little Lick Creek above SR 1814 near Oak Grove	U	U	U	U	X	U	U						
02086849	Ellerbe Creek near Gorman	U	X	X	U	X	U	U						
02087183	Neuse River near Falls	U	U	U	X	X	U	U	U	U	U	U	U	U
02087570	Neuse River at Smithfield	U	U											
02096846	Cane Creek near Orange Grove	U	U	U	X	U	U	U	U	U	U	U	U	U
02096960	Haw River near Bynum	U	U	U	X	U	U	U	X	U	U	U	U	U
02097314	New Hope Creek near Blands	U	U	U	X	U	U	U	U	U	U	U	U	U
0209741955	Northeast Creek at SR 1100 near Genlee	U	U	U	X	X	U	U	U	U	U	U	U	U
02097464	Morgan Creek near White Cross	U	U	U	X	U	U	U	U	U	U	U	U	U
02097517	Morgan Creek near Chapel Hill	U	U	U	X	U	U	U	U	U	U	U	U	U
0209782520	White Oak Creek at Green Level											X	U	U
02098198	Haw River below B. Everett Jordan dam near Moncure	U	U	U	X									

## Field Quality Control

The purpose of field quality-control sampling is to determine the effects of sample-collection and handling procedures and laboratory processes on the concentration of constituents in the stream or lake sample (hereafter referred to as the environmental sample) and to assess the replicability of results by field and laboratory processes. Field quality-control samples collected for this study consisted of blanks and replicates.

## Blank Samples

Most blank samples were equipment blanks (table 10). In addition, trip blanks (also identified as boat blanks, vehicle blanks, and truck blanks), and the USGS North Carolina District office de-ionized

**Table 10.** Numbers and types of blank samples collected for the Triangle Area Water Supply Monitoring Project in North Carolina, 1988–2001.

[USGS, U.S. Geological Survey]

Blank sample type	USGS identi- fication number	Number of samples			
		Nutrients	Metals	Major cations	Major anions
Equipment blanks					
Stream	370010100	50	31	37	6
Lake	370010101	19	14	14	0
Trip blanks					
Vehicle	370010102	17	16	16	0
Truck	10100009	1	1	1	0
Boat	10100007	4	4	4	0
Trip (unspecified)	10100004	1	1	1	0
De-ionized water supply	99000001	7	5	5	0
Total blanks		99	72	78	6



water blanks were prepared. Equipment blanks are used to test for sample contamination from collection, processing, and preservation procedures. Vehicle blanks are used to test for sample contamination from transport of sample bottles in a particular vehicle or during an entire sampling trip. The USGS District office de-ionized water blank was used to test the quality of the de-ionized water produced in the USGS North Carolina District laboratory and used for cleaning and rinsing equipment and supplies.

### De-ionized Water Blanks

Samples of de-ionized water produced in the USGS North Carolina District office were collected and analyzed to document the quality of de-ionized water that was used to clean and rinse equipment and supplies. Concentrations of major ions, trace elements, and metals in the de-ionized water typically were less than detection levels (table 11). Samples were analyzed using methods with low detection levels—less than detection levels for environmental samples.

### Equipment and Trip Blanks

For equipment blanks, de-ionized water certified by the producer as free of inorganic contamination (inorganic free) was poured through all of the clean sampling equipment, bottled, and preserved using the same procedures as those used to process and preserve stream and lake samples (hereafter, referred to as environmental samples to differentiate them from blank samples). Trip blanks were produced by pouring inorganic-free de-ionized water into a sample bottle and transporting the bottle to and from the site in a truck, boat, or both during a sampling trip.

Results of blank sample analysis were examined for inorganic constituents, total organic carbon, and color, for which environmental sample results also were analyzed (table 12). The method used for analysis of metals and trace elements in blank samples changed in 1994, resulting in analyses of total constituents and dissolved constituents. Prior to August 1994, blank samples were analyzed by using the same methods as those used for metals and trace elements in environmental samples. After August 1994, a new method with much lower detection levels for metals and trace elements was used specifically for blank samples. Results were reported for

dissolved metals and trace elements because de-ionized water contains no particulates. Results of these analyses are shown separately in table 12. With the new method, seven trace elements were analyzed in blank samples that were not analyzed in the associated environmental samples. These trace elements were antimony, barium, beryllium, boron, strontium, titanium, and uranium. Results of these analyses are listed at the end of table 12.

Results of blank sample analysis are an indication of the minimum environmental concentrations that can be measured and attributed to the environment rather than to contamination that may occur during some part of the sample collection, processing, preservation, shipping, and analysis process. The median and 90<sup>th</sup> percentile concentrations for equipment and vehicle blank samples for each constituent were calculated (table 12). The 95-percent confidence interval around the median and the upper bound of the 95-percent confidence interval of the 90<sup>th</sup> percentile were calculated by using non-parametric estimators of confidence limits for percentiles (Gilbert, 1987, p. 141). In addition, the minimum, 10<sup>th</sup> percentile, median, and 90<sup>th</sup> percentile concentrations were calculated for the pooled environmental samples (table 12).

The minimum and 10<sup>th</sup> percentile concentrations in the environmental samples were compared to the upper bound of the 95-percent confidence interval of the 90<sup>th</sup> percentile concentration for blank samples. The latter value is an indication of the maximum amount of contamination that may have been introduced to the environmental sample during collection and processing. If this value is less than the minimum environmental concentration, then contamination is not a significant factor affecting the measured environmental sample concentration.

For calcium, magnesium, sodium, and silica, the upper bound of the 95-percent confidence interval of the 90<sup>th</sup> percentile of the blank samples was at least an order of magnitude less than the 10<sup>th</sup> percentile concentration of the environmental samples. For potassium, chloride, and sulfate, the upper bound of the 95-percent confidence interval of the median of the blank samples was at or near the detection level and was at least an order of magnitude less than the 10<sup>th</sup> percentile concentration of the environmental samples. The fluoride results typically were below detection in blank and environmental samples. Sample contamination was not a factor for this group of constituents.

**Table 11.** Results of analyses of de-ionized water produced in the U.S. Geological Survey North Carolina District office.

[—, no data; E, concentration was estimated below the detection level;  $\mu\text{S}/\text{cm}$ , microsiemens per centimeter at 25 degrees Celsius;  $\text{mg}/\text{L}$ , milligram per liter; <, less than; **bold** values are above detection levels;  $\mu\text{g}/\text{L}$ , microgram per liter]

Parameter code	Constituent	Date						
		10/16/1992	2/2/1993	6/16/1993	9/30/1993	1/9/1995	1/27/1997	10/8/2002
00403	pH, laboratory, standard units	—	—	7	8.8	—	—	7.4
90095	Lab specific conductance, $\mu\text{S}/\text{cm}$	—	—	2	1	—	—	E3
00095	Field specific conductance, $\mu\text{S}/\text{cm}$	—	—	—	—	—	1	—
00915	Dissolved calcium ( $\text{mg}/\text{L}$ as Ca)	—	—	<.02	<b>0.1</b>	<b>0.003</b>	<b>0.01</b>	<.01
00925	Dissolved magnesium ( $\text{mg}/\text{L}$ as Mg)	—	—	<.01	<.01	<.001	<.001	<.008
00930	Dissolved sodium ( $\text{mg}/\text{L}$ as Na)	—	—	<.20	<.20	<.03	<b>.05</b>	<.09
00955	Dissolved silica ( $\text{mg}/\text{L}$ as $\text{SiO}_2$ )	—	—	<.01	<.01	<.02	<b>.1</b>	<.13
00608	Dissolved ammonia ( $\text{mg}/\text{L}$ as N)	—	<b>0.01</b>	<b>.003</b>	<b>.003</b>	<.002	<.002	<.015
00623	Dissolved ammonia plus organic nitrogen ( $\text{mg}/\text{L}$ as N)	—	<.20	—	—	—	—	—
00625	Total ammonia plus organic nitrogen ( $\text{mg}/\text{L}$ as N)	<0.20	<.20	—	—	—	—	—
00610	Total ammonia ( $\text{mg}/\text{L}$ as N)	<.010	—	—	—	—	—	—
00631	Dissolved nitrite plus nitrate ( $\text{mg}/\text{L}$ as N)	—	<.050	<.005	<.005	<.005	<.005	<.022
00630	Total nitrite plus nitrate ( $\text{mg}/\text{L}$ as N)	<.050	—	—	—	—	—	—
00613	Dissolved nitrite, $\text{mg}/\text{L}$ as N	—	<b>.01</b>	<.001	<b>.001</b>	<b>.001</b>	<.001	<.0023
00615	Total nitrite, $\text{mg}/\text{L}$ as N	<.010	—	—	—	—	—	—
00666	Dissolved phosphorus ( $\text{mg}/\text{L}$ as P)	—	<.010	—	—	—	—	—
00671	Dissolved orthophosphate ( $\text{mg}/\text{L}$ as P)	—	<.010	<.001	<.001	<.001	<b>.002</b>	<.0070
70507	Total orthophosphate, ( $\text{mg}/\text{L}$ as P)	<.010	—	—	—	—	—	—
00665	Total phosphorus total ( $\text{mg}/\text{L}$ as P)	<.010	<.010	—	—	—	—	—
01106	Dissolved aluminum ( $\mu\text{g}/\text{L}$ as Al)	—	—	—	—	<b>3.27</b>	<.30	<1.6
01095	Dissolved antimony ( $\mu\text{g}/\text{L}$ as Sb)	—	—	—	—	<.200	<.20	<.30
01000	Dissolved arsenic ( $\mu\text{g}/\text{L}$ as As)	—	—	—	—	—	—	<.26
01005	Dissolved barium ( $\mu\text{g}/\text{L}$ as Ba)	—	—	<2	<2	<.200	<.20	<.050
01010	Dissolved beryllium ( $\mu\text{g}/\text{L}$ as Be)	—	—	<.5	<.5	<.2	<.20	<.06
01020	Dissolved boron ( $\mu\text{g}/\text{L}$ as B)	—	—	—	—	<b>4.78</b>	<b>3.1</b>	<7
01025	Dissolved cadmium ( $\mu\text{g}/\text{L}$ as Cd)	—	—	<1.0	<1.0	<.3	<.30	<.037
01030	Dissolved chromium ( $\mu\text{g}/\text{L}$ as Cr)	—	—	<5	<5	<.200	<.20	<.8
01035	Dissolved cobalt ( $\mu\text{g}/\text{L}$ as Co)	—	—	<3	<3	<.200	<.20	<.015
01040	Dissolved copper ( $\mu\text{g}/\text{L}$ as Cu)	—	—	<10	<10	<b>0.23</b>	<b>.26</b>	<.23
01046	Dissolved iron ( $\mu\text{g}/\text{L}$ as Fe)	—	—	<3	<3	<3	<3.0	<10
01049	Dissolved lead ( $\mu\text{g}/\text{L}$ as Pb)	—	—	<10	<10	<.300	<.30	<.08
01130	Dissolved lithium ( $\mu\text{g}/\text{L}$ as Li)	—	—	<4	<4	—	—	<.50
01056	Dissolved manganese ( $\mu\text{g}/\text{L}$ as Mn)	—	—	<1	<1	<.100	<.10	<.18
01060	Dissolved molybdenum ( $\mu\text{g}/\text{L}$ as Mo)	—	—	<10	<10	<.200	<.20	<.33
01065	Dissolved nickel ( $\mu\text{g}/\text{L}$ as Ni)	—	—	<10	<10	<.500	<.50	<.06
01145	Dissolved selenium ( $\mu\text{g}/\text{L}$ as Se)	—	—	—	—	—	—	<.50
01075	Dissolved silver ( $\mu\text{g}/\text{L}$ as Ag)	—	—	<1.0	<1.0	<.2	<.20	<.20
01080	Dissolved strontium ( $\mu\text{g}/\text{L}$ as Sr)	—	—	<.500	<.500	<.100	<.10	<.20
01057	Dissolved thallium ( $\mu\text{g}/\text{L}$ as Tl)	—	—	—	—	<.100	<.10	<.041
01085	Dissolved vanadium ( $\mu\text{g}/\text{L}$ as V)	—	—	<6	<6	—	—	<.13
01090	Dissolved zinc ( $\mu\text{g}/\text{L}$ as Zn)	—	—	<b>3</b>	<3	<b>1</b>	<.50	<1
22703	Dissolved uranium, natural, water, $\mu\text{g}/\text{L}$	—	—	—	—	<.20	<.20	<.018

**Table 12.** The distribution of concentrations in equipment and trip blank samples and the minimum, median, and maximum values of environmental samples.

[mg/L, milligram per liter; <, less than; —, not enough samples to calculate; N, nitrogen; P, phosphorus; NA, no detection level established; ° C, degrees Celsius; µg/L, microgram per liter; E, value is estimated below the laboratory detection level]

Constituent	Laboratory reporting levels	Blank samples						Environmental samples				
		Number of observations	Median	90 <sup>th</sup> Per-centile	95-percent confidence level			Number of observations	Concentration			
					Lower bound of the median confidence interval	Upper bound of the median confidence interval	Upper bound of the 90 <sup>th</sup> percentile confidence interval		Minimum	10 <sup>th</sup> per-centile	Median	90 <sup>th</sup> per-centile
Major anions and cations												
Calcium, mg/L	0.002	73	0.004	0.04	0.003	0.006	0.069	1,442	1.9	4.7	6.8	9.7
Magnesium, mg/L	0.001, 0.01	73	< 0.001	0.004	< 0.001	< 0.01	0.01	1,442	0.8	1.9	2.6	3.49
Potassium, mg/L	0.1	6	< 0.1	< 0.1	< 0.1	< 0.1	—	1,442	0.1	1.4	2.4	4.2
Sodium, mg/L	0.025, 0.2, 0.03	73	< 0.03	0.041	< 0.025	< 0.03	0.18	1,442	1.2	3.7	5.6	23
Bicarbonate, mg/L	NA	6	2.55	3	1.3	3	—	1,426	5.9	18	28	41
Chloride, mg/L	0.1	6	< 0.1	0.1	< 0.1	0.1	—	1,405	0.3	4.2	6.2	16
Fluoride, mg/L	0.1	6	< 0.1	< 0.1	< 0.1	< 0.1	—	1,404	< 0.1	< 0.1	< 0.1	0.2
Silica, mg/L	0.1, 0.01, 0.02	73	< 0.02	0.316	< 0.02	< 0.02	0.388	1,383	< 0.1	4.5	9.2	14
Sulfate, mg/L	0.1	6	< 0.1	0.3	< 0.1	0.3	—	1,401	< 0.1	3	6.4	24
Nutrients, organic carbon, and physical measures												
Dissolved organic nitrogen plus ammonia, mg/L as N	0.1, 0.2	24	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	1,068	< 0.1	< 0.2	0.4	0.7
Total organic nitrogen plus ammonia, mg/L as N	0.1, 0.2	23	< 0.2	< 0.2	< 0.2	< 0.2	0.3	1,566	< 0.1	0.3	0.6	1.0
Ammonia, mg/L as N	0.002	92	< 0.002	0.02	< 0.002	0.002	0.03	1,478	< 0.002	0.009	0.04	0.14
Nitrite plus nitrate, mg/L as N	0.05, 0.005	92	< 0.005	0.005	< 0.005	< 0.005	0.008	1,499	< 0.005	< 0.05	0.23	0.95
Nitrite, mg/L as N	0.001, 0.01	92	< 0.001	< 0.003	< 0.001	< 0.001	0.01	1,220	< 0.001	< 0.01	< 0.01	0.02
Dissolved phosphorus, mg/L as P	0.01	24	< 0.01	0.01	< 0.01	< 0.01	0.02	1,093	< 0.01	< 0.01	0.02	0.128
Orthophosphate, mg/L as P	0.001, 0.01	92	0.001	0.003	< 0.001	0.001	0.008	1,478	< 0.001	< 0.01	< 0.01	0.1
Total phosphorus, mg/L as P	0.01	23	< 0.01	0.02	< 0.01	0.01	0.04	1,545	< 0.01	0.02	0.05	0.18
Total organic carbon, mg/L	NA	6	0.25	6.9	0.1	6.9	—	1,418	0.8	4.2	7.2	12
Color	1	6	3	5	1	5	—	1,417	< 1	19	45	110
Dissolved residue, evaporation at 180 °C	1	6	< 1	2	< 1	2	—	1,381	< 1	48	70	120

**Table 12.** The distribution of concentrations in equipment and trip blank samples and the minimum, median, and maximum values of environmental samples.—Continued

[mg/L, milligram per liter; <, less than; —, not enough samples to calculate; N, nitrogen; P, phosphorus; NA, no detection level established; ° C, degrees Celsius; µg/L, microgram per liter; E, value is estimated below the laboratory detection level]

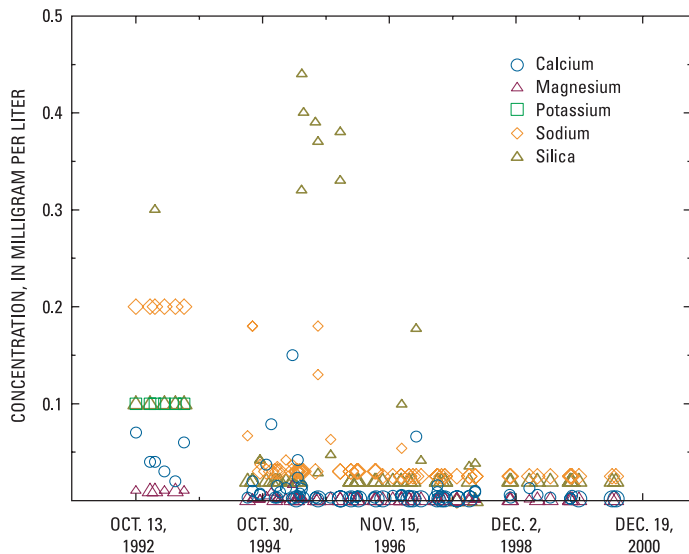
Constituent	Laboratory reporting levels	Blank samples						Environmental samples				
		Number of observations	Median	90 <sup>th</sup> Per-centile	95-percent confidence level			Number of observations	Concentration			
					Lower bound of the median confidence interval	Upper bound of the median confidence interval	Upper bound of the 90 <sup>th</sup> percentile confidence interval		Minimum	10 <sup>th</sup> per-centile	Median	90 <sup>th</sup> per-centile
Metals and trace elements												
Total aluminum, µg/L	10	22	< 10	10	< 10	< 10	40	804	< 10	40	140	1,200
Dissolved aluminum, µg/L	0.3	67	< 0.3	2.7	< 0.3	< 0.3	3.5	—	—	—	—	—
Total arsenic, µg/L	1	22	< 1	< 1	< 1	< 1	< 1	813	< 1	< 1	< 1	2
Total cadmium, µg/L	1	22	< 1	< 1	< 1	< 1	< 1	823	E0.05	< 1	< 1	< 1
Dissolved cadmium, µg/L	0.3	67	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	—	—	—	—	—
Total chromium, µg/L	1	22	< 1	< 1	< 1	< 1	6	823	E0.5	< 1	< 1	3
Dissolved chromium, µg/L	0.2	67	< 0.2	< 0.2	< 0.2	< 0.2	0.245	—	—	—	—	—
Total cobalt, µg/L	1	22	< 1	< 1	< 1	< 1	< 1	805	< 1	< 1	< 1	2
Dissolved cobalt, µg/L	0.2	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—
Total copper, µg/L	1	22	< 1	1	< 1	< 1	2	798	E0.7	< 1	2	6
Dissolved copper, µg/L	0.2	67	< 0.2	0.57	< 0.2	< 0.2	1.406	—	—	—	—	—
Total iron, µg/L	10	22	< 10	20	<10	< 10	20	963	< 10	150	580	2,300
Dissolved iron, µg/L	3	67	< 3	< 3	< 3	< 3	4.36	—	—	—	—	—
Total lead, µg/L	1	22	< 1	< 1	< 1	< 1	< 1	798	E0.6	< 1	< 1	5
Dissolved lead, µg/L	0.3	67	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	—	—	—	—	—
Total manganese, µg/L	10	22	< 10	<10	<10	< 10	< 10	962	< 10	40	130	670
Dissolved manganese, µg/L	0.1, 1	67	< 0.1	0.162	< 0.1	< 0.1	0.376	—	—	—	—	—
Total mercury, µg/L	0.1	22	< 0.1	< 0.1	< 0.1	< 0.1	0.6	815	< 0.1	< 0.1	< 0.1	< 0.1
Total molybdenum, µg/L	1	22	< 1	< 1	< 1	< 1	1	789	E0.9	< 1	< 1	2
Dissolved molybdenum, µg/L	0.2, 10	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—
Total nickel, µg/L	1	20	<1	2	<1	< 1	—	794	E0.9	< 1	< 1	3
Dissolved nickel, µg/L	0.5, 10	67	< 0.5	< 0.5	< 0.5	< 0.5	0.582	—	—	—	—	—
Total selenium, µg/L	1	22	< 1	< 1	< 1	< 1	< 1	805	< 1	< 1	< 1	< 1
Total silver, µg/L	1	22	< 1	< 1	< 1	< 1	< 1	803	< 0.4	< 1	< 1	< 1
Dissolved silver, µg/L	0.2, 1	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—
Total zinc, µg/L	10	22	<10	<10	< 10	< 10	10	807	< 10	< 10	< 10	31
Dissolved zinc, µg/L	0.5	67	0.565	2.204	< 0.5	0.71	4.813	—	—	—	—	—

**Table 12.** The distribution of concentrations in equipment and trip blank samples and the minimum, median, and maximum values of environmental samples.—Continued

[mg/L, milligram per liter; <, less than; —, not enough samples to calculate; N, nitrogen; P, phosphorus; NA, no detection level established; ° C, degrees Celsius; µg/L, microgram per liter; E, value is estimated below the laboratory detection level]

Constituent	Laboratory reporting levels	Blank samples						Environmental samples				
		Number of observations	Median	90 <sup>th</sup> Per-centile	95-percent confidence level			Number of obser-vations	Concentration			
					Lower bound of the median confi-dence interval	Upper bound of the median confi-dence interval	Upper bound of the 90 <sup>th</sup> percentile confidence interval		Minimum	10 <sup>th</sup> per-centile	Median	90 <sup>th</sup> per-centile
Metals and trace elements with no associated environmental data												
Antimony, dissolved, µg/L	0.2	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—
Barium, dissolved, µg/L	0.2	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—
Beryllium, dissolved, µg/L	0.2	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—
Boron, dissolved, µg/L	2	67	< 2	6.8	< 2	< 2	7.7	—	—	—	—	—
Strontium dissolved, µg/L	0.1	67	< 0.1	< 0.1	< 0.1	< 0.1	0.157	—	—	—	—	—
Titanium, dissolved, µg/L	0.1	67	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	—	—	—	—	—
Uranium, µg/L	0.2	67	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	—	—	—	—	—

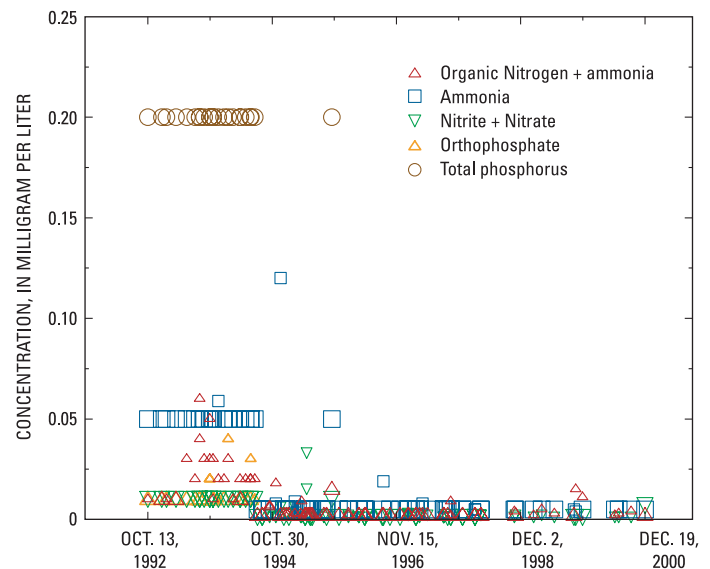
Graphs of selected major ion concentrations in blank samples over time suggest that changing to the parts-per-billion protocol (fig. 6) in 1994 probably resulted in improved sample handling and a decrease in blank sample contamination. Note that detection levels for sodium, potassium, and silica were lowered in 1993.



**Figure 6.** Concentrations of selected major ions in trip and equipment blanks during the period of study for the Triangle Area Water Supply Monitoring Project in North Carolina.

For most nutrients, the upper bound for the 95-percent confidence interval of the 90<sup>th</sup> percentile concentration of the blank samples was greater than the laboratory detection level (except for dissolved organic nitrogen plus ammonia). Furthermore, nutrient concentrations in the environmental samples were very low; for dissolved organic-plus-ammonia nitrogen, nitrite plus nitrate, nitrite, dissolved phosphorus, and orthophosphate, the 10<sup>th</sup> percentile concentration of the environmental samples was at the detection level. As a result, contamination of the environmental samples from sample collection and processing is possible but only for samples with very low concentrations. Blank concentrations over time suggest that improvements in sample handling in 1994 resulted in decreased blank sample contamination (fig. 7). Note that laboratory reporting levels for most nutrients were lowered in October 1994.

Few blank samples were analyzed for color, total organic carbon, and dissolved residue; thus, the upper bound of the



**Figure 7.** Concentrations of selected nutrients in equipment and trip blanks during the period of study for the Triangle Area Water Supply Monitoring Project in North Carolina.

95-percent confidence interval of the 90<sup>th</sup> percentile concentration could not be determined. For color and dissolved residue, the 90<sup>th</sup> percentile concentration of the blank samples was at least four times less than the 10<sup>th</sup> percentile concentration of the environmental samples. For total organic carbon, the 90<sup>th</sup> percentile concentration of the blank samples slightly exceeded the 10<sup>th</sup> percentile of the environmental samples. This indicates that contamination from sample handling may have been significant at the concentrations found in the environmental samples. Only one blank sample, however, had a concentration greater than 1.6 mg/L, and this sample was the first blank sample collected for total organic carbon in 1992.

For total metals and trace elements, 77 to 100 percent of the concentrations in the blank samples were below the laboratory reporting level (table 13); for dissolved metals and trace elements, between 46 and 100 percent were below the laboratory reporting level. The greatest potential for sample contamination (that is, the upper bound of the 95-percent confidence interval of the 90<sup>th</sup> percentile concentration for blanks was above the detection level and greater than the 10<sup>th</sup> percentile of the environmental samples) was for total aluminum, total chromium, total copper, and total mercury (table 12).

Analyses of blank samples, however, collected only after 1994 with lower reporting levels for dissolved aluminum and

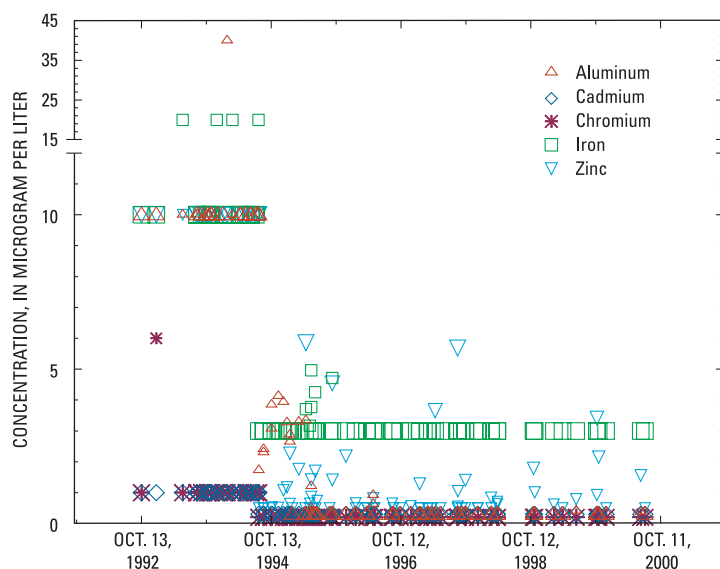
**Table 13.** The percentage of blank samples that were analyzed for metals and trace elements with concentrations above the detection level.

[µg/L, microgram per liter]

Metal or trace element	Detection level, in µg/L	Number of analyses	Analyses less than detection, in percent
Total aluminum	10	22	77
Dissolved aluminum	0.3	67	75
Total arsenic	1	22	100
Total cadmium	1	22	100
Dissolved cadmium	0.3	67	100
Total chromium	1	22	95
Dissolved chromium	0.2	67	94
Total cobalt	1	22	100
Dissolved cobalt	0.2	67	100
Total copper	1	22	82
Dissolved copper	0.2	67	73
Total iron	10	22	77
Dissolved iron	3	67	91
Total lead	1	22	100
Dissolved lead	1	22	100
Total manganese	10	22	100
Dissolved manganese	0.1	67	81
Total mercury	0.1	22	91
Total molybdenum	1	22	91
Dissolved molybdenum	0.2	67	100
Total nickel	1	20	85
Dissolved nickel	0.5	67	94
Total selenium	1	22	100
Total silver	1	22	100
Dissolved silver	0.2	67	100
Total zinc	10	22	95
Dissolved zinc	0.5	67	46

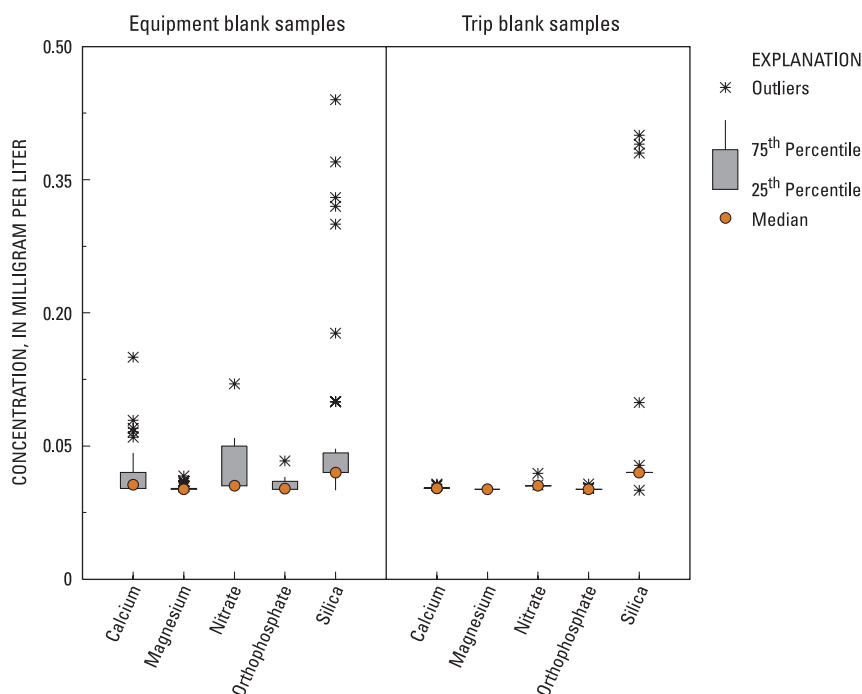
dissolved chromium indicate that contamination from sample-collection and handling procedures was not significant. For these blank samples, the upper bound of the 95-percent confidence interval of the 90<sup>th</sup> percentile concentrations for dissolved aluminum and dissolved chromium in blanks was

3.5 and 0.245 µg/L, respectively. The improvement in blank contaminant levels relative to concentrations in environmental samples is a result, in part, of the decrease in reporting levels and also may be due to the adoption of improved sample-collection and handling protocols in 1994. Likewise, iron and aluminum concentrations in blank samples have decreased during the study, and this decrease suggests that improvements in sample handling implemented in 1994 resulted in decreased blank sample contamination (fig. 8).

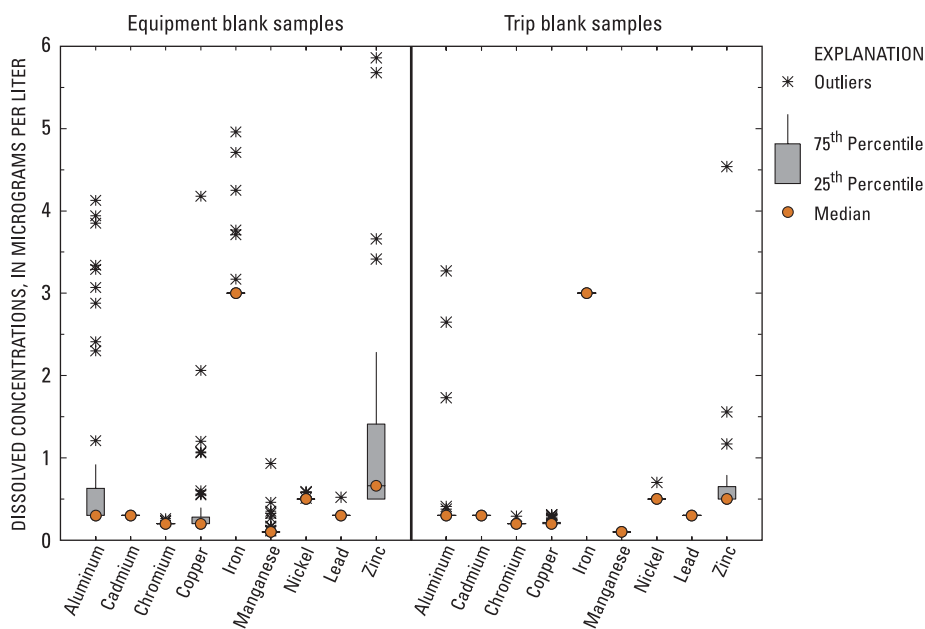


**Figure 8.** Concentrations of selected metals and trace elements in equipment and trip blanks during the period of study for the Triangle Area Water Supply Monitoring Project in North Carolina.

Concentrations in equipment blanks were compared to concentrations in trip blanks to assess whether contamination was most likely a result of sample-collection and processing procedures or procedures associated with the handling of containers during transport. For selected major ions and nutrients, contamination was most often found in equipment blanks (fig. 9). Similarly, a greater range of concentrations of selected metals and trace elements occurred in equipment blanks (fig. 10). Zinc and aluminum were exceptions to this.



**Figure 9.** Distribution of concentrations of calcium, magnesium, nitrate, orthophosphate, and silica in equipment blanks and trip blanks for the Triangle Area Water Supply Monitoring Project in North Carolina.



**Figure 10.** Distribution of dissolved concentrations of aluminum, cadmium, chromium, copper, iron, manganese, nickel, lead, and zinc in equipment blanks and trip blanks for the Triangle Area Water Supply Monitoring Project in North Carolina.



## Replicates

Two types of replicate samples were collected—split and concurrent replicate samples. Split samples were obtained by splitting a single sample into two sample bottles and assigning each a unique time of sample collection (typically 1 to 5 minutes apart). Concurrent samples were collected from the stream or lake separately but at approximately the same time. Each sample then was assigned a unique time of sample collection (typically 1 to 5 minutes apart).

Analysis of replicate sample results is used as a measure of the variability inherent in the processing and analysis of water-quality samples. Variability was calculated by determining the standard deviation and relative standard deviation of each replicate pair using the following equations:

$$s = \sqrt{\frac{(C_1 - C_2)^2}{2}}, \quad (1)$$

where

$s$  is the standard deviation of the replicates, and  $C_1$  and  $C_2$  are replicate concentrations;

$$rsd = 100 \left( \frac{s}{\bar{C}} \right), \quad (2)$$

where

$rsd$  is the relative standard deviation of the replicates,

$s$  is the standard deviation of the replicates, and

$\bar{C}$  is the average concentration.

The standard deviation of each replicate pair was compared to mean concentration of the replicate pair to assess whether variability changed with concentration. When variability was relatively constant over the range of measured concentrations, variability was expressed as standard deviation. When variability was not constant, variability was expressed as relative standard deviation.

For cadmium, mercury, selenium, and silver, all replicate concentrations were below the detection level. For arsenic, four of five concentrations that were reported as detectable were reported at the detection level (table 14). Variability could not be assessed for these constituents.

**Table 14.** Replicate variability for total metals and total trace elements.

[Concentrations are in micrograms per liter;  $\leq$ , less than or equal to;  $\pm$ , plus or minus; —, could not be computed because all concentrations were below the detection level;  $>$ , greater than;  $<$ , less than]

Constituent	Number of observations	Number above detection	Concentration range	Standard deviation or relative standard deviation	90-percent confidence interval for a result within range
Aluminum	45	45	$\leq 2,300$	20.8	$\pm 34.1$
Arsenic <sup>a</sup>	45	5	—	—	—
Cadmium	48	0	—	—	—
Chromium	48	6	$>1, <4$	0.10	$\pm 0.16$
Cobalt	45	6	$>1, <4$	0.02	$\pm 0.03$
Copper	48	40	$<6$	0.26	$\pm 0.43$
Iron	48	48	$\leq 3,300$	30.4	$\pm 49.8$
Lead	47	17	$>1$ to 5	0.11	$\pm 0.18$
Manganese	48	48	$<6,000$	17.32	$\pm 28.5$
Mercury	48	0	—	—	—
Molybdenum	45	9	$>1, <4$	0.04	$\pm 0.06$
Nickel	47	18	$>1, <4$	0.12	$\pm 0.20$
Selenium	45	0	—	—	—
Silver	48	0	—	—	—
Zinc	48	4	$>10, <40$	0.17	$\pm 0.29$

<sup>a</sup>All but one arsenic concentration were at the detection level.

For total organic carbon and chlorophyll *a* and *b*, standard deviation was relatively constant at low ranges but increased with increasing concentration at high ranges. For these constituents, variability is expressed as a relative concentration, in percent, for the high ranges (table 15).

The 90-percent confidence interval for a result, shown in tables 14 and 15, indicate the accuracy of a particular result within range. These data can be used to assess whether differences in results for environmental data, either at different sites or at different times at the same site, are significant.

## Laboratory Quality Assurance and Quality Control

The general quality-assurance practices used by the NWQL are described in Friedman and Erdmann (1982), Pritt and Raese (1992), and Pirkey and Glodt (1998). Three elements compose quality control at the NWQL—method performance, data review and blind sample programs, and performance-evaluation studies. Quality-control samples to determine method performance are prepared and analyzed by the chemist. The chemist uses these data to control the analytical process for each batch of environmental samples. If acceptance criteria are not met, then the analysis is discontinued until the problem is corrected. The NWQL Quality Assurance Section provides data review and monitors long-term method performance throughout the laboratory. Results from the blind sample programs, described below, also are used in monitoring long-term method performance. Finally, the NWQL participates in performance-evaluation studies managed by external agencies to compare NWQL performance to other laboratories.

## Method-Performance Checks

Laboratory blank, standard, and replicate samples are among the tools used to monitor and correct the performance of an analytical method. For inorganic analyses, laboratory quality control for each sample set consists of blanks, calibration standards, standard reference samples, and replicate environmental samples. The laboratory blank is prepared from water free of inorganic constituents being measured. Calibration standards are prepared and analyzed at a range of concentrations similar to the range expected in the environmental samples. Standard reference samples are samples of known concentration that are used to assess bias for each analysis and are used for assessment of long-term method performance.

For organic analysis, laboratory quality control for each sample set consists of laboratory reagent blanks, reagent spikes, and surrogate compounds. The laboratory reagent blank is

prepared with water that is known to be free of the compounds being analyzed. If, in a sample set, a compound is detected in the blank, the source of contamination is sought and the set of samples is reanalyzed. If the source is not determined, then the detection level for the analyte is increased to the concentration of the reagent blank. The laboratory reagent spike has a known concentration of compounds of interest. The results of analysis are compared with acceptance criteria for the analysis to assess potential bias. Results also are tracked on a long-term basis to monitor long-term trends in bias and variability. A surrogate is a compound that is not expected to be in the environmental sample but on which the method performs similarly. When added to each environmental and quality-control sample, a surrogate is used to assess recovery of the compound. Paper copies of results of blank, spike, and surrogate analyses for sample sets that included project samples were provided by the laboratory and stored in project files.

## Blind Sample Program

The NWQL has two inorganic blind sample programs. One is an internally administered program to quantify bias from laboratory contamination. The Inorganic Blind Sample Project (IBSP) of the USGS Branch of Quality Systems provides independent, external quality assurance for the NWQL. The Branch of Quality Systems produces double-blind quality-control samples that are submitted to the laboratory through USGS District offices. A double-blind sample is a sample for which the sample origin and constituent concentration of the sample are unknown to the analyst.

Samples are made from USGS standard reference samples to appear to the laboratory analyst to be as much like environmental samples as possible. They are subject to the same laboratory handling, processing, and analytical procedures as samples collected for the Triangle Area Water Supply Monitoring Project. The Branch of Quality Systems evaluates the analytical results by comparing the value reported by the laboratory to the most probable value (MPV) for the IBSP sample. The MPV is the median of results for each constituent reported by participants in a round-robin evaluation of USGS standard reference samples. The NWQL results are considered acceptable if they are within 2 standard deviations of the MPV.

Quality-control data from the IBSP can be used to assess laboratory error during specific periods of time. Most importantly, the information is used to assist the laboratory in detecting and correcting problems in analytical procedures (U.S. Geological Survey, 1997–present). Monthly IBSP reports are provided to USGS offices regarding NWQL performance based on blind-sample results. In addition, the IBSP reports include the corrective actions taken by the laboratory.

**Table 15.** Replicate variability for physical measurements, major ions, nutrients, and chlorophyll.

[Concentrations are in milligrams per liter unless otherwise noted; <, less than; ±, plus or minus; µS/cm, microsiemens per centimeter at 25 degrees Celsius; ≤, less than or equal to; >, greater than; ANC as CaCO<sub>3</sub>, acid neutralizing capacity as calcium carbonate; NH<sub>4</sub>, ammonia; N, nitrogen; TKN, organic nitrogen plus ammonia; NO<sub>2</sub>, nitrite; NO<sub>3</sub>, nitrate; P, phosphorus; PO<sub>4</sub>, orthophosphate; µg/L, microgram per liter]

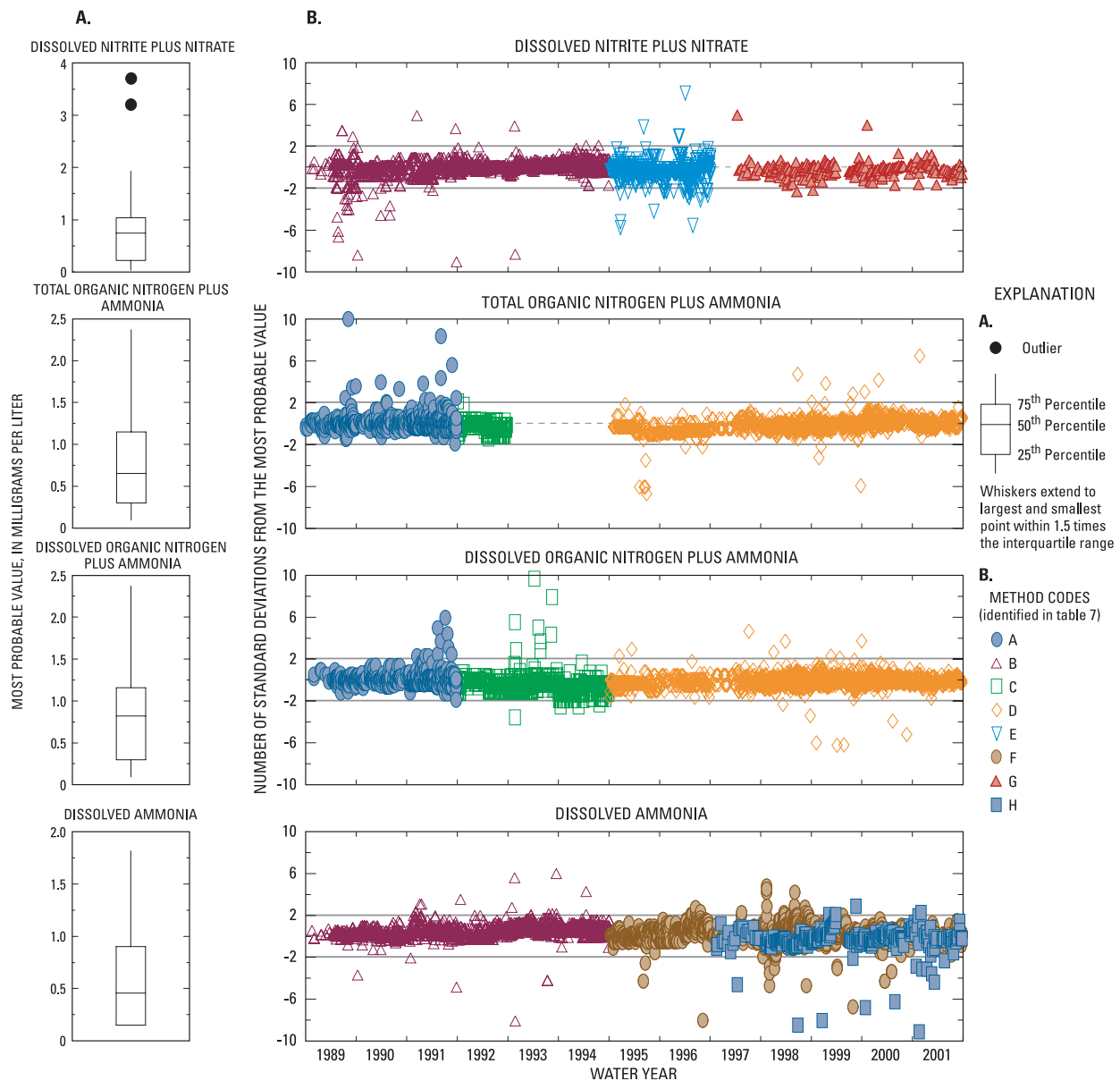
Constituent	Number of observations	Number above detection	Concentration range	Standard deviation or relative standard deviation	90-percent confidence interval for a result within range
Laboratory pH, standard units	96	96	< 10	0.10	±0.16
Specific conductance, µS/cm	96	96	< 500	0.78	±1.3
Calcium	96	96	< 30	0.08	±0.13
Magnesium	96	96	< 6	0.04	±0.06
Potassium	96	96	< 18	0.06	±0.10
Sodium	75	75	≤ 10	0.06	±0.10
	21	21	> 10 to 65	0.38	±0.62
ANC as CaCO <sub>3</sub>	96	96	< 50	0.27	±0.24
Chloride	92	92	≤ 44	0.16	±0.26
Fluoride	92	49	< 0.4	0.01	±0.02
Silica	89	89	< 20	0.24	±0.39
Sulfate	92	92	< 100	0.17	±0.28
Dissolved NH <sub>4</sub> as N	94	78	< 3.0	0.004	±0.006
Total NH <sub>4</sub> as N	25	22	< 3.0	0.004	±0.006
Dissolved TKN as N	78	70	< 2.0	0.05	±0.08
Total TKN as N	95	85	< 2.0	0.06	±0.10
Dissolved NO <sub>2</sub> +NO <sub>3</sub> as N	94	72	< 3.0	0.02	±0.03
Total NO <sub>2</sub> +NO <sub>3</sub> as N	25	18	< 3.0	0.02	±0.03
Dissolved NO <sub>2</sub> as N	82	41	< 0.12	0.001	±0.002
Total NO <sub>2</sub> as N	24	9	< 0.06	0.002	±0.003
Total phosphorus as P	94	84	< 0.6	0.01	±0.02
Dissolved phosphorus as P	78	47	< 0.5	0.01	±0.02
Dissolved PO <sub>4</sub> as P	91	53	≤ 0.5	0.004	±0.006
Total PO <sub>4</sub> as P	25	15	< 0.5	0.01	±0.02
Total organic carbon <sup>a</sup>	80	80	≤9.0	0.30	±0.49
	13	13	9.0 to 20	26%	±43.3%
Color	59	59	≤ 60	3.6	±5.9
	34	34	> 60, < 150	11.6	±19.1
Residue on evaporation	92	92	< 300	6.6	±10.8
Chlorophyll <i>a</i> <sup>a</sup> , µg/L	14	14	≤10	1.1	±1.8
	6	6	10 to 40	14.2%	±23.3%
Chlorophyll <i>b</i> <sup>a</sup> , µg/L	13	11	≤ 0.3	0.07	0.12
	7	7	> 0.3, < 2.1	8.1%	±13.3%

<sup>a</sup>Standard deviation for total organic carbon and chlorophyll *a* and *b* was relatively constant at low ranges but increased with increasing concentrations at high ranges; thus, variability is expressed as a relative concentration in percent (%) for the high ranges.

Control charts are presented to assess the accuracy (variability) and bias of individual constituent analyses during the period of the project (for example, fig. 11). Changes in symbol color on control charts indicate a change in the method of analysis for a particular constituent. A cross reference for method codes (single alpha characters) is provided in table 7. The solid lines at positive and negative standard deviations of 2 indicate the analytical results that meet the laboratory quality-control criterion. The distribution of the constituent

concentration of blind-sample submissions also is shown (fig. 11).

The performance of methods for total and dissolved organic nitrogen plus ammonia, ammonia, and nitrite plus nitrate (fig. 11) are shown in scatterplots. Analyses for total and dissolved organic nitrogen plus ammonia were somewhat variable and positively biased in the early 1990's; however, the number of analyses more than two standard deviations from the MPV were small. The number of analyses outside of two



**Figure 11.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of dissolved nitrite plus nitrate, total and dissolved organic nitrogen plus ammonia, and dissolved ammonia for water years 1989–2001.

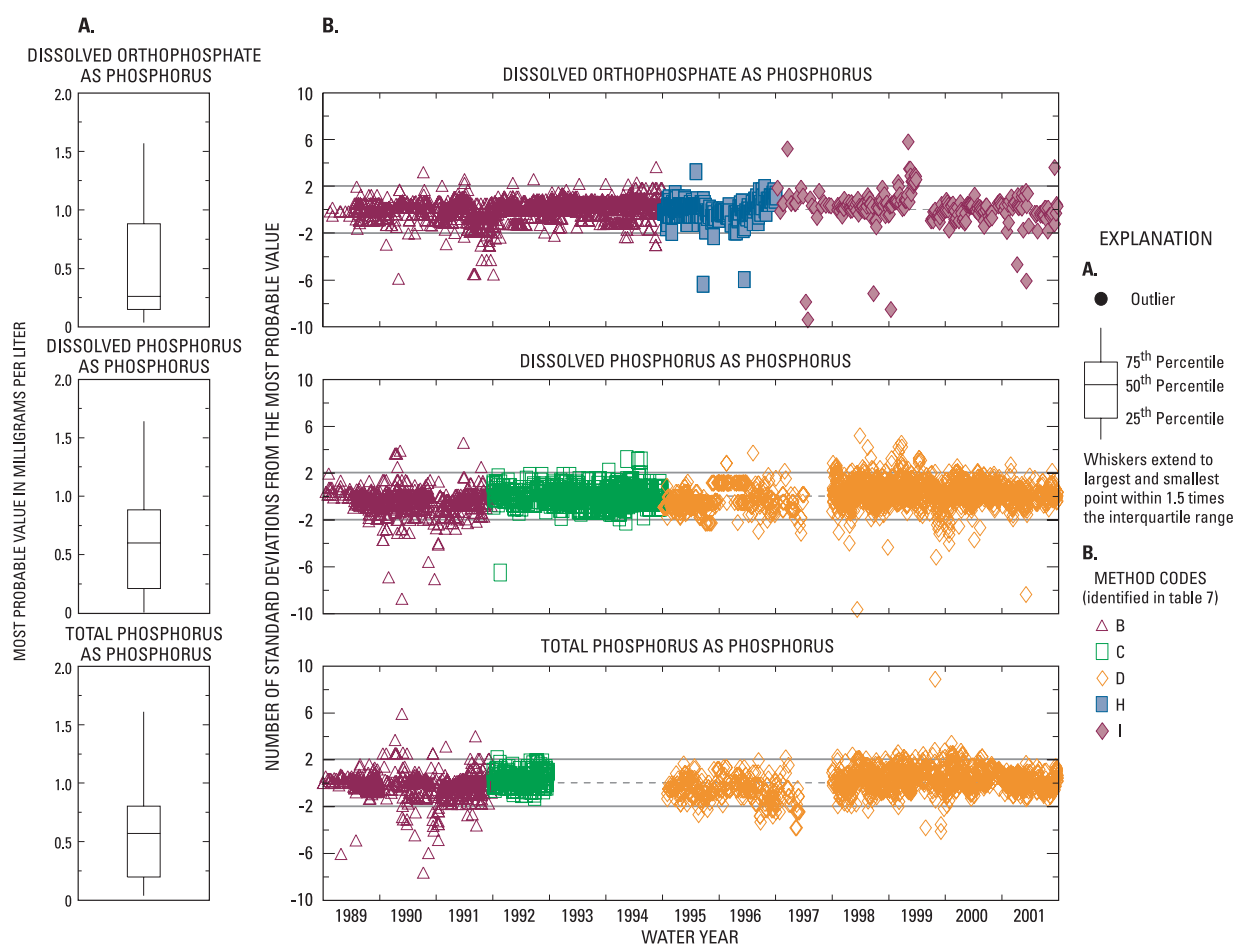
standard deviations from the MPV was greater for ammonia, which was variable throughout the study. Nitrite data (not shown) are available only through 1991. Nitrite plus nitrate analyses were variable in the first 2 years of study and improved until a method change in 1995. Another method change in 1997 brought improved precision.

Total and dissolved phosphorus were most variable in the first 3 years of the study (fig. 12). A method change in 1992 increased the precision of analyses. Dissolved orthophosphate analyses also were most variable in the first 3 years and improved thereafter with the exception of several significant outliers after 1995.

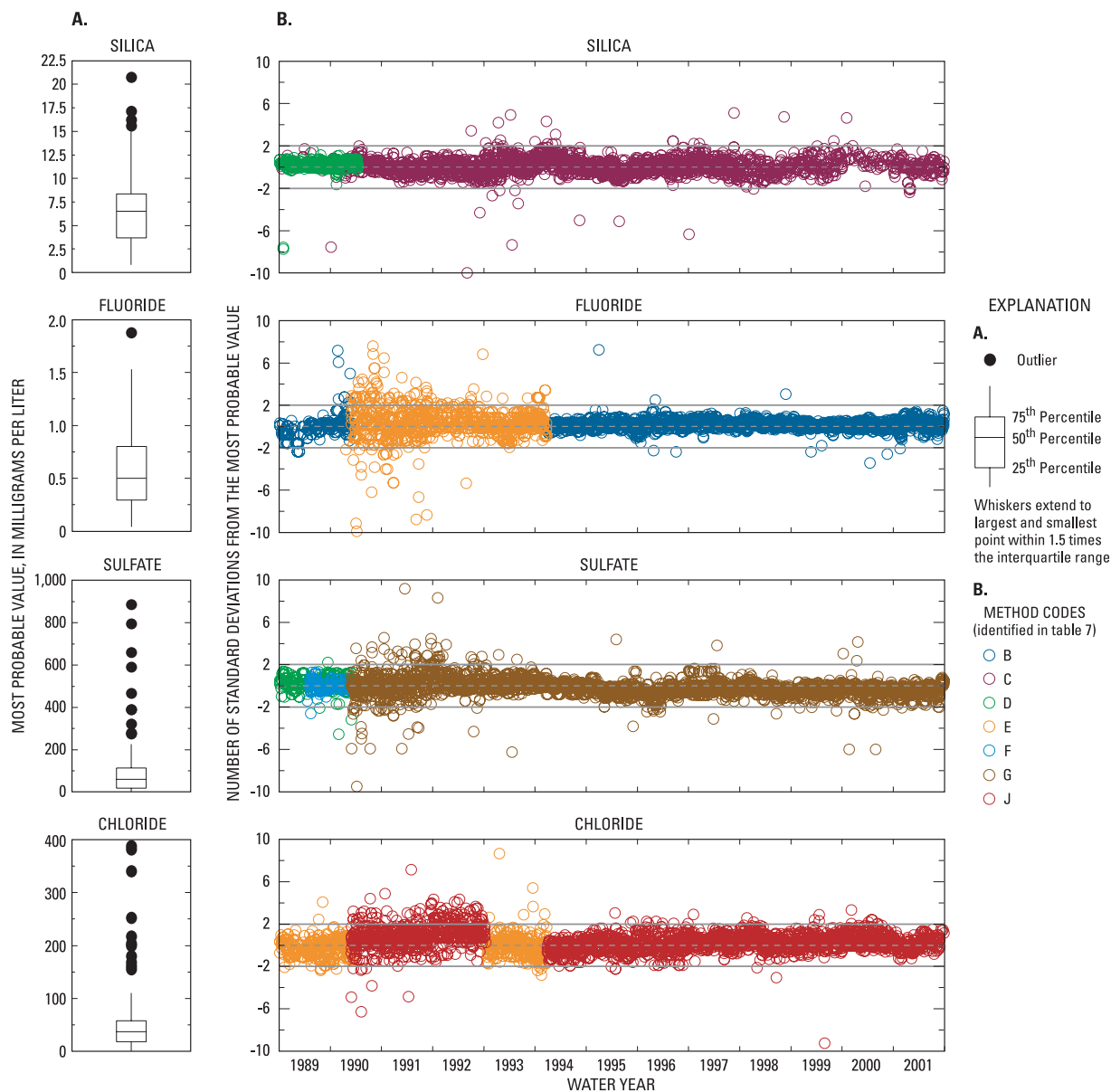
Analyses of silica, fluoride, sulfate, and chloride (fig. 13); and sodium, magnesium, and calcium (fig. 14) were unbiased with low variability over the period of study. Variability of fluoride, sulfate, and chloride increased during 1990 through 1992 when a new analytical method was used. Analyses of potassium also were unbiased over the period of study but had greater variability (fig. 14).

Control charts and distributions of blind samples for metals and trace elements are shown in figures 15, 16, and 17. Most are unbiased and many have very low variability over the period of study (for example, cobalt, chromium, cadmium (fig. 15), nickel, molybdenum, lead (fig. 16), selenium, and silver (fig. 17)). Analyses of iron were highly variable and biased high for the first half of the study (fig. 16). Likewise, manganese analyses, although not as variable, were biased high prior to the 1993 water year. Zinc analyses were biased somewhat high between water years 1989 and 1994 (fig. 17). Analyses are unbiased thereafter and are very stable with the method change that occurred in 1999 (table 7). Likewise, aluminum, iron, and manganese analyses were variable during the first 5 years of study and have become markedly more stable since a method change in 1999. Mercury, which was somewhat biased from about 1996 to 1999, has been more stable since 1999.

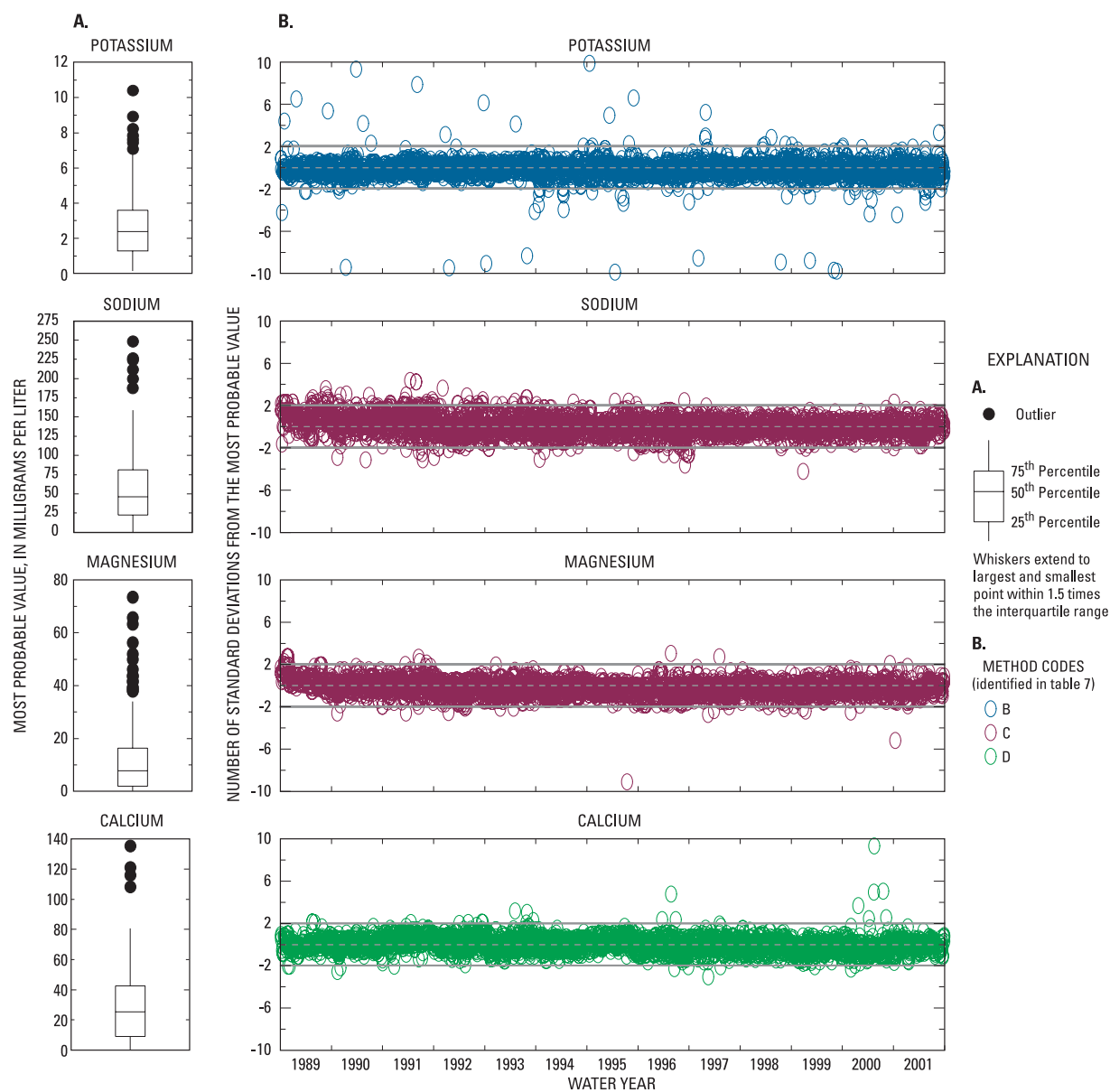
The Organic Blind Sample Project (OBSP) of the USGS Branch of Quality Systems provides independent, external



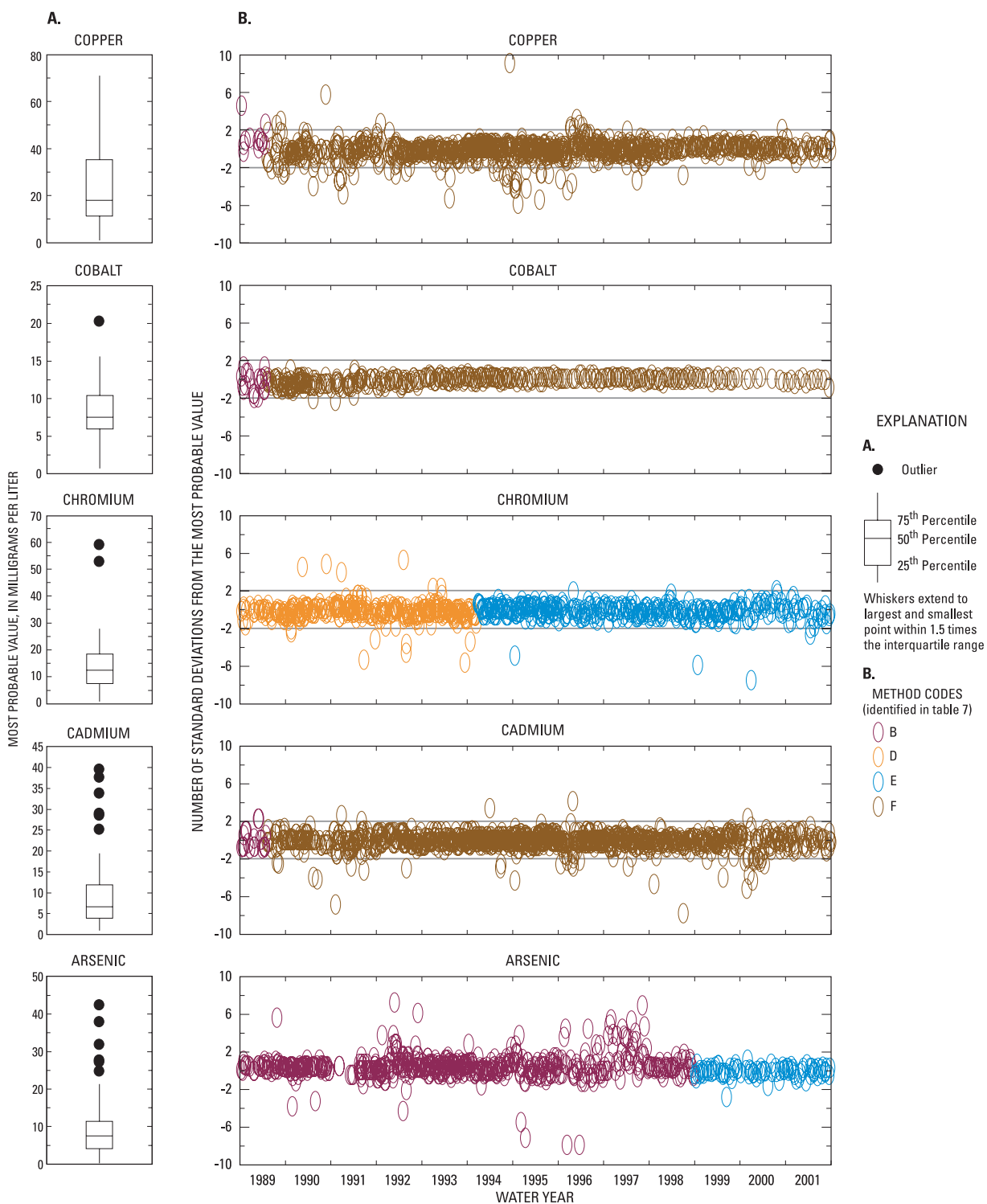
**Figure 12.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of dissolved orthophosphate and total and dissolved phosphorus for water years 1989–2001.



**Figure 13.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of silica, fluoride, sulfate, and chloride for water years 1989–2001.

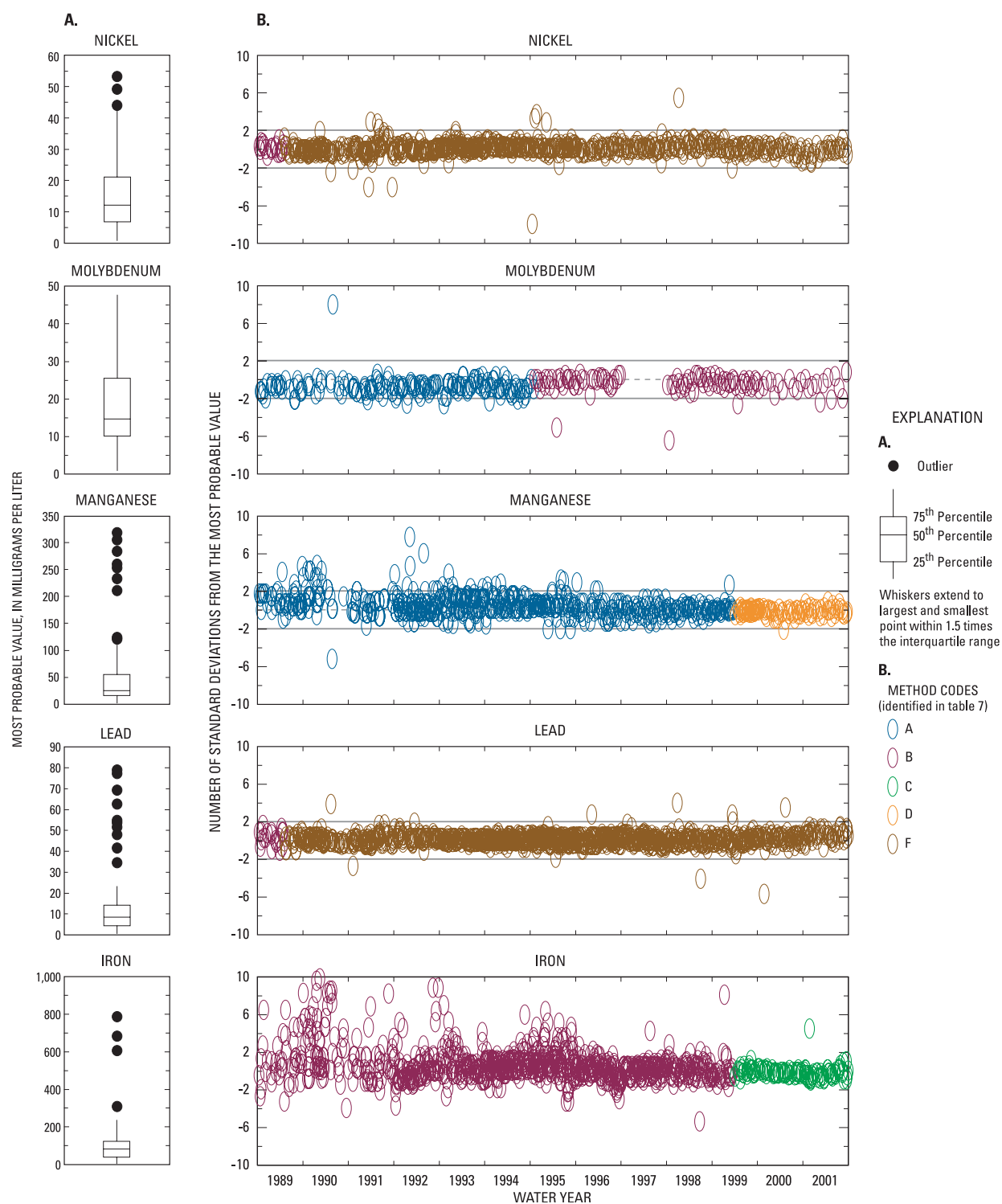


**Figure 14.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of potassium, sodium, magnesium, and calcium for water years 1989–2001.

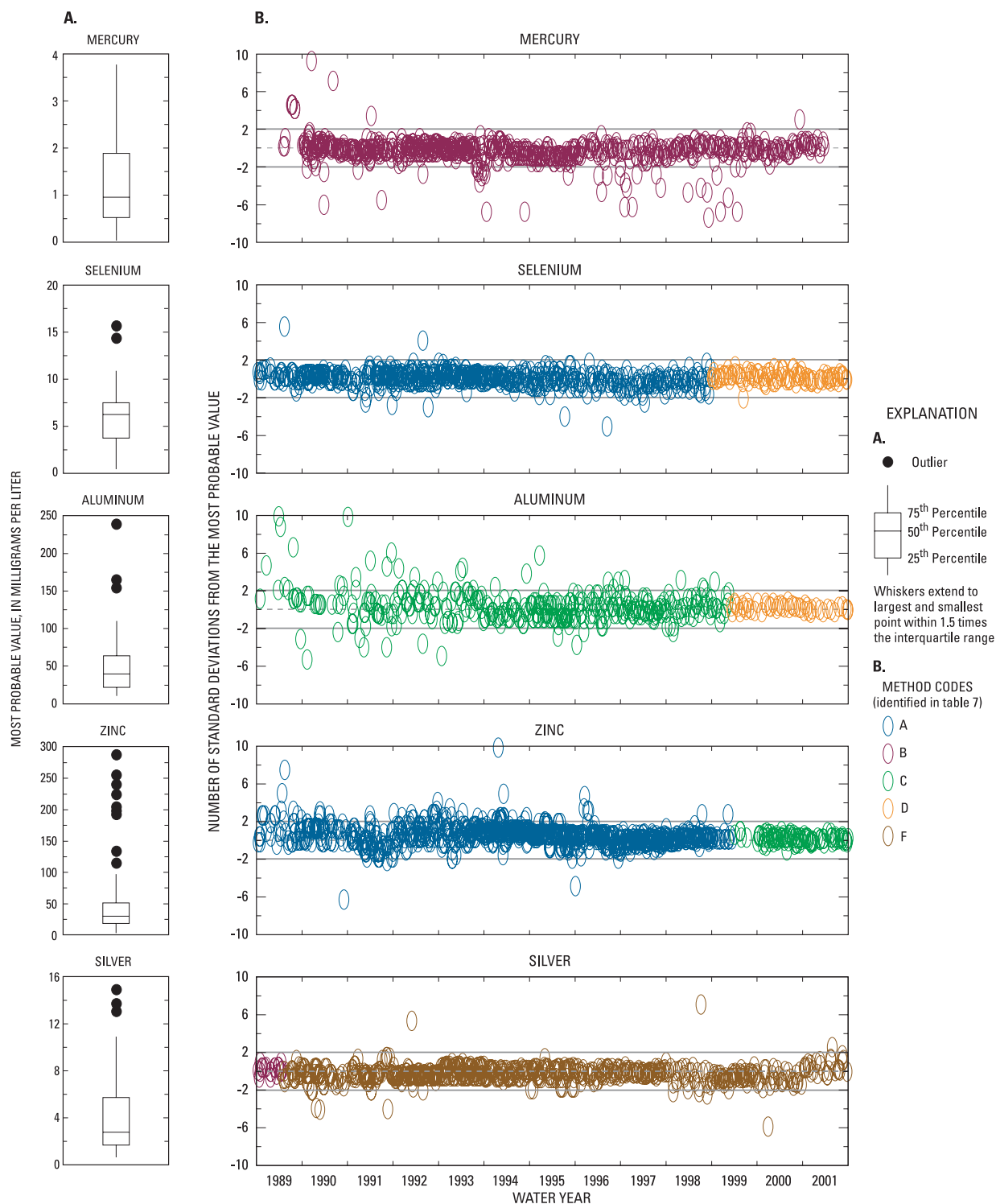


**Figure 15.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of copper, cobalt, chromium, cadmium, and arsenic for water years 1989–2001.





**Figure 16.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of nickel, molybdenum, manganese, lead, and iron for water years 1989–2001.



**Figure 17.** National Water Quality Laboratory control charts from the U.S. Geological Survey Inorganic Blind Sample Project showing (A) the distribution of double-blind sample concentrations and (B) trends in deviations from double-blind sample concentrations of mercury, selenium, aluminum, zinc, and silver for water years 1989–2001.

quality assurance for organic analyses performed at the NWQL. The OBSP produces double-blind samples of known composition that are submitted to the laboratory. Spike mixtures used to produce the quality-control samples are purchased from qualified manufacturers. As of this publication date, limited OBSP quality-control data are available online at <http://btdqs.usgs.gov/OBSP/index.html> and, beginning in January 1996, can be used to assess laboratory bias and variability. The USGS Branch of Quality Systems compiles the data and provides reports on data quality (trends in bias, variability, and errors) to the NWQL on a quarterly basis so that corrective actions can be taken.

## Data Review

The NWQL Quality Assurance Section (QAS) has responsibility for final review of analyses before they are reported to the user. Chemical logic checks, such as anion-cation balance, specific conductance-anion ratio, specific conductance-cation ratio, dissolved solids-specific conductance ratio, field and laboratory measurement agreement for pH, alkalinity, and specific conductance, and filtered components compared with raw components of constituents, are automatically processed by computer. Analytical results that do not meet acceptance criteria are reviewed by chemists in the QAS, and samples either are reanalyzed or results are approved.

## Performance-Evaluation Studies

The NWQL participates in national and international interlaboratory performance-evaluation studies and has performed very well (Pirkey and Glodt, 1998). These performance-evaluation studies are as follows:

- U.S. Environmental Protection Agency water pollution, water supply, and safe drinking water act studies;
- USGS Branch of Quality Systems evaluation program for standard reference samples;
- National Water Research Institute of Canada; and
- National Oceanic and Atmospheric Administration intercomparison program for marine sediment and tissue.

## Summary

The Triangle Area Water Supply Monitoring Project was initiated in October 1988 to provide long-term water-quality data for six area water-supply reservoirs and their tributaries. Additional objectives of the project are to provide data that can be used to determine the effectiveness of large-scale changes in water-resource management practices, document differences in

water quality among water-supply types (large multiuse reservoir, small reservoir, run of river), and provide tributary-loading and in-lake data for water-quality modeling of Falls and Jordan Lakes. By September 2001, the project had progressed in four phases and included as many as 34 sites. Most sites were sampled and analyzed by the USGS. Some sites were already part of the North Carolina DWQ statewide ambient water-quality network and continued to be sampled for that network. The DWQ sites were sampled by the USGS only occasionally during high-flow events to supplement data collected by the DWQ. The network has provided data on streamflow, physical properties, and concentrations of nutrients, major ions, metals, trace elements, chlorophyll *a* and *b*, total organic carbon, suspended sediment, and selected synthetic organic compounds.

Each study phase had a special area of focus for which extra targeted sampling was done. In phase I, the special focus was on the occurrence of synthetic organic compounds in streambed sediments in the Haw River basin. In phase II the special focus was to identify whether pesticides detected in phase I in streamwater downstream from wastewater discharges originated from the wastewater effluents, from upstream sources, or both. In phase III, the special focus was on the presence of *Cryptosporidium parvum* oocysts and *Giardia Lamblia* cysts in raw-water supplies. In phase IV, the special focus was on the collection of more samples during high-flow events.

Records of water quality collected by the USGS were stored in the NWIS database. Environmental water-quality data can be retrieved from the USGS Website <http://waterdata.usgs.gov/nwis/> and also are included in the appendix of this report. Quality-control data can be obtained by contacting the USGS North Carolina District office in Raleigh. Methods of sample collection follow standard published and unpublished USGS protocols. A significant change in data-collection methods occurred in 1992 when the parts-per-billion protocol became the standard protocol for the USGS. These protocols are detailed in the USGS National Field Manual.

Some laboratory methods and reporting levels changed during the study. A method reference for each constituent is stored in NWIS with the water-quality record. Reporting levels for each constituent and method are reported herein.

Instantaneous streamflow at the time of sample collection was obtained from records of continuous gage height at streamgaging stations in the network. Streamflow records were stored in the NWIS database and can be retrieved from the USGS North Carolina District office in Raleigh. A goal of the project was to measure water quality during a range of streamflow conditions. A comparison of long-term streamflow durations to sampled streamflow durations indicates that this goal was achieved, and samples were collected over a wide range of streamflow conditions, including during many high-flow events.

Project quality-assurance activities include written procedures for sample collection, record management and archive, collection of field quality-control samples (blank

samples and replicate samples), and monitoring of the quality of field supplies. In addition to project quality-assurance activities, the quality of laboratory analyses was assessed through laboratory quality-assurance practices and an independent laboratory quality-control assessment provided by the USGS Branch of Quality Systems through the Inorganic Blind Sample Project (IBSP) and the Organic Blind Sample Project (OBSP).

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**Table 7.** Inorganic constituents and physical properties analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analyses.

[USGS, U.S. Geological Survey; ID, identification; mg/L, milligram per liter; ICP, induction-coupled argon plasma; **bold** numbers are lowest reporting level; °C, degree Celsius; —, not applicable; µS/cm, micro-siemens per centimeter at 25 °C; std, standard; CaCO<sub>3</sub>, calcium carbonate; PCU, platinum cobalt units; µg/L, microgram per liter; MBAS, methylene blue active substances; N, nitrogen; ASF, automated segmented flow; P, phosphorus; OES, optical emission spectrometry; AA, atomic absorption spectrometry]

Constituent	Parameter code	Method code	Method reference <sup>a</sup>	USGS method ID <sup>b</sup>	Period of record	Reporting level	Analytical method
<b>Major ions</b>							
Calcium, in mg/L	00915	D	B, D	I-1472-87	10/88 – 09/95	0.1	Atomic emission spectrometric, ICP.
					10/95 – 09/98	0.02	
					10/98 – 10/00	<b>0.02</b>	
					10/00 – 09/01	<b>0.011</b>	
Magnesium, in mg/L	00925	C	B, D	I-1472-87	10/88 – 09/95	0.1	Atomic emission spectrometric, ICP.
					10/95 – 12/97	0.01	
					12/97 – 09/98	0.004	
					10/98 – 09/99	<b>0.004</b>	
					10/99 – 10/00	<b>0.014</b>	
					10/00 – 09/01	<b>0.008</b>	
Sodium, in mg/L	00930	C	B, D	I-1472-87	10/88 – 09/95	0.1	Atomic emission spectrometric, ICP.
					10/95 – 12/97	0.2	
					12/97 – 09/98	0.1	
					10/98 – 09/99	<b>0.06</b>	
					10/99 – 10/01	<b>0.09</b>	
Potassium, in mg/L	00935B	B	E	I-1630-85	10/88 – 09/99	0.1	Atomic absorption spectrometry, direct.
					10/99 – 10/00	<b>0.24</b>	
					11/00 – 09/01	<b>0.11</b>	
Chloride, in mg/L	00940	E	E	I-2187-78	10/88 – 04/90	0.1	Colorimetry, automated.
	00940	J	B	I-2057-90	04/90 – 10/92	0.1	Ion-exchange chromatography, OMNIPAX-500.
	00940	E	E	I-2057-85	12/92 – 01/94	0.1	Colorimetry, automated.
	00940	J	E	I-2057-85	1/3/94 – 09/99	0.1	Ion-exchange chromatography, Dionex AS4A.
					10/99 – 11/00	<b>0.29</b>	
					11/00 – 09/01	<b>0.08</b>	
Sulfate, in mg/L	00945	D	E	I-2823-85	10/88 – 04/89	0.2	Turbidimetry, automated.
	00945	F	E	I-2823-85	04/89 – 04/90		Turbidimetry, automated (corrected).
	00945	G	B	I-2057-90	04/90 – 12/93	0.1	Ion-exchange chromatography, OMNIPAX-500.
			E	I-2057-85	01/94 – 09/99	0.1	
					10/99 – 11/00	<b>0.31</b>	Ion-exchange chromatography, Dionex AS4A.
					11/00 – 09/01	<b>0.11</b>	

**Table 7.** Inorganic constituents and physical properties analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analyses.—Continued

[USGS, U.S. Geological Survey; ID, identification; mg/L, milligram per liter; ICP, induction-coupled argon plasma; **bold** numbers are lowest reporting level; °C, degree Celsius; —, not applicable; µS/cm, micro-siemens per centimeter at 25 °C; std, standard; CaCO<sub>3</sub>, calcium carbonate; PCU, platinum cobalt units; µg/L, microgram per liter; MBAS, methylene blue active substances; N, nitrogen; ASF, automated segmented flow; P, phosphorus; OES, optical emission spectrometry; AA, atomic absorption spectrometry]

Constituent	Parameter code	Method code	Method reference <sup>a</sup>	USGS method ID <sup>b</sup>	Period of record	Reporting level	Analytical method
Major ions (Continued)							
Fluoride, in mg/L	00950	B	E	I-2327-78	10/88 – 04/90	0.1	Ion selective electrode, automated.
	00950	E	B	I-2057-90	04/90 – 12/93	0.1	Ion-exchange chromatography, OMNIPAX-500.
	00950	B	E	I-2327-85	01/94 – 10/00	0.1	Ion selective electrode, automated.
					10/00 – 09/01	<b>0.16</b>	
Silica, in mg/L	00955	D	B	I-1472-87	10/88 – 04/90	0.1	Atomic emission spectrometry, ICP.
	00955	C	E	I-2700-85	04/90 – 10/00	0.1	Colorimetry, automated, molybdate blue.
					10/00 – 09/01	<b>0.48</b>	
Physical properties and general water quality							
Temperature in °C	00010	—	—	—	10/88 – 09/01	0.5	Thermometer/thermistor.
Specific conductance, field, in µS/cm	00095	—	—	—	10/88 – 09/01	None	Electrometric, Wheatstone bridge.
Specific conductance, laboratory, in µS/cm	90095	A	E	I-2781-85	10/88 – 09/01	1	Electrometric, Wheatstone bridge.
pH, field, in std units	00400	—	—	—	10/88 – 09/01	None	Electrometric, ion-selective electrode.
pH, laboratory, in std units	00403	A	E	I-2587-89	10/88 – 09/01	0.1	Electrometric, ion-selective electrode.
Acid neutralizing capacity (ANC), in mg/L as CaCO <sub>3</sub>	Various		E	I-2030-89	10/88 – 09/01	None	Electrometric/titration to pH 4.5.
Bicarbonate, in mg/L as CaCO <sub>3</sub>	Various		—	—	10/88 – 09/01	None	Electrometric/incremental titration.
Dissolved oxygen, in mg/L	00300	—	—	—	10/88 – 09/01	0.1	Electrometric.
Total organic carbon, in mg/L	00680	A	F	O-3100-83	10/88 – 09/01	0.1	Wet oxidation.
Dissolved solids, in mg/L	70300	A	E	I-1750-89	10/88 – 12/97	1	Evaporation at 180 °C, gravimetric.
					12/97 – 09/01	10	
Color, in PCU	00080	A	E	I-1250-89	10/88 – 09/01	1	Electrometric, visual comparison.
Chlorophyll <i>a</i> , phytoplankton, in µg/L	70953	A	F	B-6530-85	10/88 – 09/01	0.1	High pressure liquid-chromatography, spectrophotometric.
Chlorophyll <i>b</i> , phytoplankton, in µg/L	70954	A	F	B-6530-85	10/88 – 09/01	0.1	High pressure liquid-chromatography, spectrophotometric.
MBAS, in mg/L	38260	A	F	O-3111-83	10/88 – 10/90	0.01	Colorimetry.
Suspended sediment, in mg/L	80154	—			10/88 – 10/01	1.0	Gravimetric.



**Table 7.** Inorganic constituents and physical properties analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analyses.—Continued

[USGS, U.S. Geological Survey; ID, identification; mg/L, milligram per liter; ICP, induction-coupled argon plasma; **bold** numbers are lowest reporting level; °C, degree Celsius; —, not applicable; µS/cm, micro-siemens per centimeter at 25 °C; std, standard; CaCO<sub>3</sub>, calcium carbonate; PCU, platinum cobalt units; µg/L, microgram per liter; MBAS, methylene blue active substances; N, nitrogen; ASF, automated segmented flow; P, phosphorus; OES, optical emission spectrometry; AA, atomic absorption spectrometry]

Constituent	Parameter code	Method code	Method reference <sup>a</sup>	USGS method ID <sup>b</sup>	Period of record	Reporting level	Analytical method
<b>Nutrients</b>							
Total ammonia plus organic nitrogen, in mg/L as N	00625	A	E	I-4552-85	10/88 – 10/91	0.1	Kjeldahl block digestion, salicylate-hypochlorite ASF, colorimetry.
	00625	C	G	I-4515-91	10/91 – 09/94	0.2	Micro-Kjeldahl digestion, ASF, colorimetry.
	00625	D	G, I	I-4515-91	10/94 – 10/97	0.2	Micro-Kjeldahl digestion, ASF, colorimetry, acidified, new field preservative
					11/97 – 10/00 10/00 – 09/01	<b>0.1</b> <b>0.08</b>	
Dissolved ammonia plus organic nitrogen, in mg/L as N	00623	A	E	I-2552-85	10/88 – 10/91	0.1	Kjeldahl block digestion salicylate-hypochlorite, ASF, colorimetry.
	00623	C	G	I-2515-91	10/91 – 09/94	0.2	Micro-Kjeldahl digestion, ASF, colorimetry.
	00623	D	G, I	I-2515-91	10/94 – 11/97	0.2	Micro-Kjeldahl digestion, ASF, colorimetry, acidified, new field preservative
					10/97 – 09/98 10/98 – 09/01	0.1 <b>0.1</b>	
Total ammonia, in mg/L	00610	B	E	I-4523-85	10/88 – 09/92	0.01	Colorimetry, ASF, salicylate-hypochlorite (method discontinued 10/92).
Dissolved ammonia, in mg/L as N	00608	B	B	I-2522-90	10/88 – 10/94	0.01	Colorimetry, ASF, salicylate-hypochlorite.
	00608	F	B, I	I-2522-90	10/94 – 11/97	0.015	Colorimetry, ASF, salicylate-hypochlorite, new field preservative
					12/97 – 10/00 10/00 – 09/01	0.02 <b>0.041</b>	
	00608	H	B	I-2525-89	10/96 – 09/01	<b>0.002</b>	Colorimetry, ASF, salicylate-hypochlorite, low-level.
Total nitrite, in mg/L as N	00615	B	E	I-4540-85	10/88 – 09/92	0.01	Colorimetry, automated segmented flow, diazotization (method discontinued 10/92).
Dissolved nitrite, in mg/L as N	00613	B	B	I-2340-90	10/90 – 10/94	0.01	Colorimetry, diazotization, ASF.
	00613	F	B, I	I-2340-90	10/94 – 09/00 10/00 – 10/01	0.01 0.006	Colorimetry, diazotization, ASF, new field preservative
	00613	H	B	I-2542-89	10/96 – 09/01	0.001	Colorimetry, diazotization, ASF, low-level.
Total nitrite plus nitrate, in mg/L as N	00630	B	E	I-4545-90	10/88 – 09/92	0.1	Cadmium reduction-diazotization, ASF, colorimetry (method discontinued 10/92).
Dissolved nitrite plus nitrate, in mg/L as N	00631	B	B	I-2545-90	10/88 – 09/91 10/91 – 09/94	0.1 0.05	Cadmium reduction-diazotization, ASF, colorimetry.
	00631	E	B, I	I-2545-90	10/94 – 09/96	0.05	Cadmium reduction-diazotization, ASF, colorimetry, new field preservative
	00631	G	B	I-2546-91	10/96 – 09/01	0.005	Cadmium reduction-diazotization, ASF, colorimetry, low-level.

**Table 7.** Inorganic constituents and physical properties analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analyses.—Continued

[USGS, U.S. Geological Survey; ID, identification; mg/L, milligram per liter; ICP, induction-coupled argon plasma; **bold** numbers are lowest reporting level; °C, degree Celsius; —, not applicable; µS/cm, microsiemens per centimeter at 25 °C; std, standard; CaCO<sub>3</sub>, calcium carbonate; PCU, platinum cobalt units; µg/L, microgram per liter; MBAS, methylene blue active substances; N, nitrogen; ASF, automated segmented flow; P, phosphorus; OES, optical emission spectrometry; AA, atomic absorption spectrometry]

Constituent	Parameter code	Method code	Method reference <sup>a</sup>	USGS method ID <sup>b</sup>	Period of record	Reporting level	Analytical method
<b>Nutrients (Continued)</b>							
Total phosphorus, in mg/L as P	00665	B	E	I-4600-85	10/88 – 09/91	0.01	Kjeldahl persulfate digestion, ascorbic acid reduction, colorimetry.
	00665	C	H	I-4610-91	10/91 – 09/94	0.01	Micro-Kjeldahl digestion, ASF, colorimetry.
	00665	D	H, I	I-4610-91	10/94 – 09/98	0.01	Micro-Kjeldahl digestion, ASF, colorimetry, acidified, new field preservative
					10/98 – 09/00	<b>0.05</b>	
					10/00 – 09/01	<b>0.06</b>	
Dissolved phosphorus, in mg/L as P	00666	B	E	I-2600-85	10/88 – 09/91	0.01	Kjeldahl persulfate digestion, ascorbic acid reduction, colorimetry.
	00666	C	H	I-2610-91	10/91 – 10/94	0.01	Micro-Kjeldahl digestion, ASF, colorimetry.
	00666	D	H, I	I-2610-91	10/94 – 09/96	0.01	Micro-Kjeldahl digestion, ASF, colorimetry, new field preservative
Total orthophosphorus, mg/L as P	70507	A	E	I-4601-85	10/88 – 11/92	0.01	Ascorbic acid reduction, ASF, colorimetry (method discontinued 10/92).
Dissolved orthophosphorus, in mg/L as P	00671	B	B	I-2601-90	10/88 – 10/94	0.01	Colorimetry, phosphomolybdate, ASF.
	00671	H	B, I	I-2601-90	10/94 – 09/96	0.01	Colorimetry, phosphomolybdate, ASF, new field preservative
	00671	I	B	I-2606-89	10/96 – 09/00	0.001	Colorimetry, phosphomolybdate, ASF, low-level.
					10/00 – 09/01	<b>0.007</b>	
<b>Metals and trace elements</b>							
Aluminum, in µg/L	01105	C	E	I-3054-86	10/88 – 12/98	10	Digestion, atomic emission spectrometry, direct current plasma.
	01105	D	A	I-4471-97	01/99 – 09/01	<b>28</b>	AA, ICP-OES.
Arsenic, in µg/L	01002	B	E	I-4062-85	10/88 – 09/98	1	Digestion, hydride generation, AA.
	01002	E	J	I-4063-98	10/98 – 10/00	<b>2.6</b>	Digestion, graphite furnace AA.
					11/00 – 09/01	<b>1.9</b>	
Cadmium, in µg/L	01027B	B	E	I-3136-85	10/88 – 05/89	1.0	Digestion, chelation extraction, AA.
	01027	F	B	I-2138-89	05/89 – 09/99	1.0	Digestion, graphite furnace, AA.
					10/99 – 09/01	<b>0.11</b>	
Chromium, in µg/L	01034	D	E	I-3229-87	10/88 – 01/94	1.0	Digestion, atomic emission spectrometry, d-c plasma.
	01034	E	C	I-3233-93	01/94 – 09/99	1.0	Digestion, graphite furnace, AA.
					10/99 – 09/01	<b>1.0</b>	

**Table 7.** Inorganic constituents and physical properties analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analyses.—Continued

[USGS, U.S. Geological Survey; ID, identification; mg/L, milligram per liter; ICP, induction-coupled argon plasma; **bold** numbers are lowest reporting level; °C, degree Celsius; —, not applicable; µS/cm, micro-siemens per centimeter at 25 °C; std, standard; CaCO<sub>3</sub>, calcium carbonate; PCU, platinum cobalt units; µg/L, microgram per liter; MBAS, methylene blue active substances; N, nitrogen; ASF, automated segmented flow; P, phosphorus; OES, optical emission spectrometry; AA, atomic absorption spectrometry]

Constituent	Parameter code	Method code	Method reference <sup>a</sup>	USGS method ID <sup>b</sup>	Period of record	Reporting level	Analytical method
<b>Metals and trace elements (Continued)</b>							
Cobalt, in µg/L	01037	B	E	I-3240-85	10/88 – 05/89	1.0	Digestion, chelation extraction, AA.
	01037	F	B	I-4243-89	05/89 – 09/99	1.0	Digestion, graphite furnace, AA.
					10/99 – 09/00	<b>1.8</b>	
					10/00 – 05/01	<b>2.4</b>	
					05/01 – 09/01	<b>1.8</b>	
Copper, in µg/L	01042	B	E	I-3271-85	10/88 – 5/89	1.0	Digestion, chelation extraction, AA.
	01042	F	B	I-4274-89	6/89 – 9/99	1.0	Digestion, graphite furnace, AA.
					10/99 – 9/00	<b>1.2</b>	
					10/00 – 5/01	<b>1.8</b>	
					5/01 – 9/01	<b>1.2</b>	
Iron, in µg/L	01045	B	E	I-3381-85	11/88 – 11/98	10	Digestion, direct aspiration, AA.
	01045	C	A	I-4471-97	3/99 – 9/01	<b>14</b>	Atomic emission spectrometry, ICP-OES.
Lead, in µg/L	01051	B	E	I-3400-85	10/88 – 5/89	5.0	Digestion, chelation extraction, AA.
	01051	F	B	I-4403-89	5/89 – 9/99	1	Digestion, graphite furnace, AA.
					10/99 – 9/01	<b>1</b>	
Manganese, in µg/L	01055	A	E	I-3454-85	11/88 – 11/98	10	Digestion, direct aspiration, AA.
	01055	D	A	I-4471-97	3/99 – 9/99	<b>3</b>	Atomic emission spectrometric, ICP-OES.
					10/99 – 9/01	<b>2.8</b>	
Mercury, in µg/L	71900	B	E	I-3462-85	10/88 – 9/99	0.1	Cold vapor, flameless, AA.
					10/99 – 10/00	<b>0.3</b>	
					10/00 – 3/01	<b>0.14</b>	
	71900	D	K	I-4464-01	4/01 – 9/01	0.01	Cold vapor, atomic fluorescence.
Molybdenum, in µg/L	01062	A	E	I-3490-85	11/88 – 10/94	1.0	Digestion, chelation extraction, flame, AA.
	01062	B	L	I-3492-96	11/94 – 10/00	1.0	Digestion, graphite furnace, AA.
					10/00 – 9/01	<b>1.5</b>	
Nickel, in µg/L	01067	B	E	I-3500-85	10/88 – 5/89	1.0	Digestion, chelation extraction, AA.
	01067	F	B	I-4503-89	5/89 – 9/99	1.0	Digestion, graphite furnace, AA.
					10/99 – 9/01	<b>1.8</b>	

**Table 7.** Inorganic constituents and physical properties analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analyses.—Continued

[USGS, U.S. Geological Survey; ID, identification; mg/L, milligram per liter; ICP, induction-coupled argon plasma; **bold** numbers are lowest reporting level; °C, degree Celsius; —, not applicable; µS/cm, micro-siemens per centimeter at 25 °C; std, standard; CaCO<sub>3</sub>, calcium carbonate; PCU, platinum cobalt units; µg/L, microgram per liter; MBAS, methylene blue active substances; N, nitrogen; ASF, automated segmented flow; P, phosphorus; OES, optical emission spectrometry; AA, atomic absorption spectrometry]

Constituent	Parameter code	Method code	Method reference <sup>a</sup>	USGS method ID <sup>b</sup>	Period of record	Reporting level	Analytical method
<b>Metals and trace elements (Continued)</b>							
Selenium, in µg/L	01147	A	E	I-4667-85	11/88 – 9/98	1.0	Digestion, hydride conversion, AA.
	01147	D	J	I-4668-98	10/98 – 9/99	1.0	Digestion, graphite furnace, AA.
					10/99 – 9/01	<b>2.6</b>	
Silver, in µg/L	01077	B	E	I-3720-85	10/88 – 5/89	1.0	Digestion, chelation extraction, AA.
	01077	F	B	I-4724-89	5/89 – 10/00	1.0	Digestion, graphite furnace, AA.
					10/00 – 9/01	<b>0.43</b>	
Zinc, in µg/L	01092	A	E	I-3900-85	11/88 – 11/98	10	Digestion, direct aspiration, AA.
	01092	C	A	I-4471-97	12/98– 9/99	<b>40</b>	Digestion, atomic emission spectrometric, ICP-OES.
					10/99 – 9/01	<b>31</b>	

<sup>a</sup>Method references: (A) Garbarino and Struzeski, 1998; (B) Fishman, 1993; (C) McLain, 1993; (D) Faires, 1993; (E) Fishman and Friedman, 1989; (F) Wershaw and others, 1987; (G) Patton and Truitt, 2000; (H) Patton and Truitt, 1992; (I) U.S. Geological Survey, 1994; (J) Jones and Garbarino, 1999; (K) Garbarino and Damrau, 2001; (L) Jones and McLain, 1997.

<sup>b</sup>USGS analytical method identification number.

**Table 8.** Synthetic organic constituents analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analysis.

[µg/L, microgram per liter; GC/MS, gas chromatography/mass spectrometry; P&T-GC/MS, purge and trap gas chromatography/mass spectrometry; PCNs, polychlorinated naphthalenes; PCBs, polychlorinated biphenyls; GC/ECD, gas chromatography/electron-capture detector; GC/FPD, gas chromatography/flame-photometric detector; —, not applicable]

Class of organic compound	Method reference <sup>a</sup> and notes	Constituent name	Parameter code	Method code	Analytical methods	Reporting level, in µg/L
Acid and base/neutral extractable semi-volatile organic compounds	O-3116-87 (Fishman, 1993). Sampled three times per year during 1989–90 water years; used as a follow-up analysis after 1990 for selected samples scanned using gas chromatography/flame ionization detector (GC/FID)	Acenaphthylene	34200	A	GC/MS	5
		Acenaphthene	34205	A	GC/MS	5
		Anthracene	34220	A	GC/MS	5
		Benzo(B)fluoranthene	34230	A	GC/MS	10
		Benzo(K)fluoranthene	34242	A	GC/MS	10
		Benzo(A)pyrene	34247	A	GC/MS	10
		bis 2-chloroethyl ether	34273	A	GC/MS	5
		bis (2-chloroethoxy) methane	34278	A	GC/MS	5
		bis (2-chloroisopropyl) ether	34283	A	GC/MS	5
		N-butylbenzylphthalate	34292	A	GC/MS	
		Chrysene	34320	A	GC/MS	
		Diethyl phthalate	34336	A	GC/MS	
		Dimethyl phthalate	34341	A	GC/MS	
		Fluoranthene	34376	A	GC/MS	
		Fluorene	34381	A	GC/MS	
		Hexachlorocyclopentadiene	34386	A	GC/MS	
		Hexachloroethane	34396	A	GC/MS	
		Indeno(1,2,3-cd)pyrene	34403	A	GC/MS	10
		Isophorone	34408	A	GC/MS	5
		N-nitrosodi-N-propylamine	34428	A	GC/MS	5
		N-Nitrosodiphenylamine	34433	A	GC/MS	5
		N-nitrosodimethylamine	34438	A	GC/MS	5
		Nitrobenzene	34447	A	GC/MS	5
		para chlorometa cresol			GC/MS	30
		Phenanthrene	34461	A	GC/MS	5
		Pyrene	34247	A	GC/MS	5
		Benzo(g,h,i)perylene	34521	A	GC/MS	10
		Benzo(a)anthracene	34526	A	GC/MS	10
		1,2-Dichlorobenzene	34536	A	GC/MS	5
		1,2,4-Trichlorobenzene	34551	A	GC/MS	5
		(1,2-5,6)Dibenzanthracene	34556	A	GC/MS	10
		1,3-Dichlorobenzene	34566	A	GC/MS	5
		1,4-Dichlorobenzene	34571	A	GC/MS	5
		2-Chloronaphthalene	34581	A	GC/MS	5
		2-Chlorophenol	34586	A	GC/MS	5
		2-Nitrophenol	34591	A	GC/MS	5
		Di-N-octylphthalate	34596	A	GC/MS	10
		2,4-Dichlorophenol	34601	A	GC/MS	5
		2,4-Dimethylphenol	34606	A	GC/MS	5
		2,4-Dinitrotoluene	34611	A	GC/MS	5
		2,4-Dinitrophenol	34616	A	GC/MS	20
		2,4,6-Trichlorophenol	34621	A	GC/MS	20
		2,6-Dinitrotoluene	34626	A	GC/MS	5

**Table 8.** Synthetic organic constituents analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analysis.—Continued

[µg/L, microgram per liter; GC/MS, gas chromatography/mass spectrometry; P&T-GC/MS, purge and trap gas chromatography/mass spectrometry; PCNs, polychlorinated naphthalenes; PCBs, polychlorinated biphenyls; GC/ECD, gas chromatography/electron-capture detector; GC/FPD, gas chromatography/flame-photometric detector; —, not applicable]

Class of organic compound	Method reference <sup>a</sup> and notes	Constituent name	Parameter code	Method code	Analytical methods	Reporting level, in µg/L
		3,3-Dichlorobenzidine	34631	A	GC/MS	20
		4-Bromophenyl ether	34636	A	GC/MS	5
		4-Chlorophenyl ether (new)	34641	B	GC/MS	5
		4-Nitrophenol	34646		GC/MS	30
		4,6-Dinitro-orthocresol	0-3		GC/MS	30
		Phenol	34694	A	GC/MS	5
		naphthalene	34696	A	GC/MS	5
		Pentachlorophenol	39047	A	GC/MS	30
		bis(2-Ethylhexyl)phthalate	39100	A	GC/MS	5
		Di-N-butyl phthalate	39110	A	GC/MS	5
		Benzidine (new)	39120	A	GC/MS	40
		Hexachlorobenzene	39700	A	GC/MS	5
		Hexachlorobutadiene	39702	A	GC/MS	5
		1,2-Diphenylhydrazine	82626	A	GC/MS	5
Volatile organic compounds	O-3127-94, schedule 1380 (Rose and Schroeder, 1995). Collected in the 1989 – 91 water years	Dichlorobromomethane	32101	A	P&T-GC/MS	0.2
			32101	B		
			32101	C		
	O-4127-96, schedule 2091 (Connor and others, 1997). Collected in the 1998 – 2001 water years	Carbon tetrachloride	32102	B	P&T-GC/MS	0.2
		1,2-Dichloroethane	32103	B	P&T-GC/MS	0.2
		Bromoform	32104	A	P&T-GC/MS	0.2
			32104	B		
			32104	C		
		Chlorodibromomethane	32105	B	P&T-GC/MS	0.2
		Chloroform	32106	B	P&T-GC/MS	0.2
		Toluene	34010	B	P&T-GC/MS	0.2
		Benzene	34030	B	P&T-GC/MS	0.2
		Chlorobenzene	34301	B	P&T-GC/MS	0.2
		Chloroethane	34311	B	P&T-GC/MS	0.2
		Ethylbenzene	34371	B	P&T-GC/MS	0.2
		Methylbromide	34413	B	P&T-GC/MS	0.2
		Chloromethane (methyl chloride)	34418	B	P&T-GC/MS	0.2
		Methylene chloride	34423	B	P&T-GC/MS	0.2
		Tetrachloroethylene	34475	B	P&T-GC/MS	0.2
		Trichlorofluoromethane	34488	B	P&T-GC/MS	0.2
		1,1-Dichloroethane	34496	B	P&T-GC/MS	0.2
		1,1-Dichloroethylene	34501	B	P&T-GC/MS	0.2
		1,1,1-Trichloroethane	34506	B	P&T-GC/MS	0.2
		1,1,2-Trichloroethane	34511	B	P&T-GC/MS	0.2
		1,1,2,2-Tetrachloroethane	34516	B	P&T-GC/MS	0.2
		o-Dichlorobenzene(1,2-Dichlorobenzene)	34536	B	P&T-GC/MS	0.2
		1,2-Dichloropropane	34541	B	P&T-GC/MS	0.2
		1,2 Trans dichloroethylene	34546	B	P&T-GC/MS	0.2
		1,3-Dichloropropene	34561	B	P&T-GC/MS	0.2

**Table 8.** Synthetic organic constituents analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analysis.—Continued

[µg/L, microgram per liter; GC/MS, gas chromatography/mass spectrometry; P&T-GC/MS, purge and trap gas chromatography/mass spectrometry; PCNs, polychlorinated naphthalenes; PCBs, polychlorinated biphenyls; GC/ECD, gas chromatography/electron-capture detector; GC/FPD, gas chromatography/flame-photometric detector; —, not applicable]

Class of organic compound	Method reference <sup>a</sup> and notes	Constituent name	Parameter code	Method code	Analytical methods	Reporting level, in µg/L
		1,3-Dichlorobenzene (m-Dichlorobenzene)	34566	B	P&T-GC/MS	0.2
		1,4-Dichlorobenzene (p-Dichlorobenzene)	34571	B	P&T-GC/MS	0.2
		2-Chloroethylvinyl ether	34576	B	P&T-GC/MS	0.2
		Dichlorodifluoromethane	34668	B	P&T-GC/MS	0.2
		Trans 1,3 Dichloropropene	34699	B	P&T-GC/MS	0.2
		Cis 1,3 Dichloropropene	34704	B	P&T-GC/MS	0.2
		Vinyl chloride	39175	B	P&T-GC/MS	0.2
		Trichloroethylene	39180	B	P&T-GC/MS	0.2
		Styrene	77128	B	P&T-GC/MS	0.2
		1,2-Dibromomethane	77651	B	P&T-GC/MS	0.2
		Xylene	81551	B	P&T-GC/MS	0.2
Organochlorine and organophosphorus pesticides plus gross PCBs, schedules 1319, 1324, 1399	O-3104-83 (Wershaw and others, 1987). Collected in water years 1989 – 94	Perthane, total	39034	A	GC/ECD	0.100
		Gross PCNs, total	39250	A	GC/ECD	0.100
		Aldrin, total	39330	B	GC/ECD	0.01
		Lindane, total	39340	B	GC/ECD	0.01
		Chlordane, total	39350	B	GC/ECD	0.100
		DDD, total	39360	B	GC/ECD	0.01
		DDE, total	39365	B	GC/ECD	0.01
		DDT, total	39370	B	GC/ECD	0.01
		Dieldrin, total	39380	B	GC/ECD	0.01
		Endosulfan, total	39388	B	GC/ECD	0.01
		Endrin, total	39390	B	GC/ECD	0.01
		Ethion, total	39398	B	GC/FPD	0.010
		Toxaphene, total	39400	B	GC/ECD	1.000
		Heptachlor, total	39410	B	GC/ECD	0.01
		Heptachlor epoxide, total	39420	B	GC/ECD	0.01
		Methoxychlor, total	39480	B	GC/ECD	0.010
		Gross PCBs, total	39516	B	GC/ECD	0.100
		Malathion, total	39530	B	GC/FPD	0.010
		Parathion, total	39540	B	GC/FPD	0.010
		Diazinon, total	39570	B	GC/FPD	0.010
		Methylparathion, total	39600	B	GC/FPD	0.010
		Mirex, total	39755	B	GC/ECD	0.010
		Carbophenothion, total	39786	B	GC/FPD	0.010
		Methyltrithion, total	39790	B	GC/FPD	0.010
		Chlorpyrifos, total	38932	A	GC/FPD	0.010
		Disyston, total (Disulfoton)			GC/FPD	0.010
		Phorate, total	39023	A	GC/FPD	0.010
		Tribuphos, total	39040	A	GC/FPD	0.010
		Fonofos(dyfonate), total	82614	C	GC/FPD	0.010

**Table 8.** Synthetic organic constituents analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analysis.—Continued

[µg/L, microgram per liter; GC/MS, gas chromatography/mass spectrometry; P&T-GC/MS, purge and trap gas chromatography/mass spectrometry; PCNs, polychlorinated naphthalenes; PCBs, polychlorinated biphenyls; GC/ECD, gas chromatography/electron-capture detector; GC/FPD, gas chromatography/flame-photometric detector; —, not applicable]

Class of organic compound	Method reference <sup>a</sup> and notes	Constituent name	Parameter code	Method code	Analytical methods	Reporting level, in µg/L
Organochlorine and organophosphorus pesticides plus gross PCBs, schedule 1334	O-3104-83 (Wershaw and others, 1987). Collected in water years 1989 – 91	Aldrin	39330	C	—	0.013
		S,S,S-tributylphosphorothioate	39040	A	—	0.02
		Chlordane, technical mix	39350	B	—	0.1
		Chlorpyrifos	38932	A	—	0.014
		Diazinon	39570	B	—	0.02
		Dieldrin	39380	C	—	0.017
		Disulfoton	39011	A	—	0.1
		alpha-Endosulfan	39388	C	—	0.015
		Endrin	39390	C	—	0.023
		Ethion	39398	B	—	0.013
		Fonofos	82614	C	—	0.012
		Heptachlor	39410	C	—	0.014
		Heptachlor epoxide	39420	C	—	0.009
		Lindane	39340	C	—	0.014
		Malathion	39530	B	—	0.1
		p,p'-Methoxychlor	39480	B	—	0.015
		Parathion-methyl	39600	B	—	0.015
		Mirex	39755	B	—	0.012
		p,p'-DDD	39360	C	—	0.016
		p,p'-DDE	39365	C	—	0.014
		p,p'-DDT	39370	C	—	0.009
		Parathion	39540	B	—	0.012
		Polychlorinated biphenyls	39516	B	—	0.1
		Phorate	39023	A	—	0.019
		Toxaphene	39400	B	—	1
		Carbophenothion	39786	B	—	0.021
Organonitrogen pesticides, filtered schedule 1379	O-1121-91 (Sandstrom and others, 1992), collected in the 1992 – 93 water years	Alachlor, dissolved	46342	A	SPE-GC/MS <sup>b</sup>	0.05
		Ametryn, dissolved	38401	A	SPE-GC/MS <sup>b</sup>	0.05
		Atrazine, dissolved	39632	A	SPE-GC/MS <sup>b</sup>	0.05
		Deisopropyl atrazine, dissolved	04038	A	SPE-GC/MS <sup>b</sup>	0.05
		Deethylatrazine, dissolved	04040	A	SPE-GC/MS <sup>b</sup>	0.05
		Metolachlor, dissolved	39415	A	SPE-GC/MS <sup>b</sup>	0.05
		Metribuzin, dissolved	82630	A	SPE-GC/MS <sup>b</sup>	0.05
		Prometryn, dissolved	04037	A	SPE-GC/MS <sup>b</sup>	0.05
		Propazine, dissolved	38535	A	SPE-GC/MS <sup>b</sup>	0.05
		Simazine, dissolved	04035	A	SPE-GC/MS <sup>b</sup>	0.05
Pesticides, filtered schedule 2001	O-1126-95 (Zaugg and others, 1995), collected in the 1994 – 2001 water years	2,6-Diethylaniline	82660	D	SPE/GCMS <sup>c</sup>	0.0017
		Acetochlor	49260	D	SPE/GCMS <sup>c</sup>	0.0041
		Alachlor	46342	D	SPE/GCMS <sup>c</sup>	0.0024
		alpha-hexachlorocyclohexane	34253	D	SPE/GCMS <sup>c</sup>	0.0046
		Atrazine	39632	D	SPE/GCMS <sup>c</sup>	0.007
		Azinphos-methyl	82686	D	SPE/GCMS <sup>c</sup>	0.05



**Table 8.** Synthetic organic constituents analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analysis.—Continued

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Class of organic compound	Method reference <sup>a</sup> and notes	Constituent name	Parameter code	Method code	Analytical methods	Reporting level, in µg/L
		Benfluralin	82673	D	SPE/GCMS <sup>c</sup>	0.010
		Butylate	04028	D	SPE/GCMS <sup>c</sup>	0.002
		Carbaryl	82680	D	SPE/GCMS <sup>c</sup>	0.041
		Carbofuran	82674	D	SPE/GCMS <sup>c</sup>	0.020
		Chlorpyrifos	38933	D	SPE/GCMS <sup>c</sup>	0.005
		cis-Permethrin	82687	D	SPE/GCMS <sup>c</sup>	0.006
		Cyanazine	04041	D	SPE/GCMS <sup>c</sup>	0.018
		Dacthal (DCPA)	82682	D	SPE/GCMS <sup>c</sup>	0.0030
		Deethylatrazine	04040	D	SPE/GCMS <sup>c</sup>	0.006
		Diazinon	39572	D	SPE/GCMS <sup>c</sup>	0.005
		Dieldrin	39381	D	SPE/GCMS <sup>c</sup>	0.0048
		Disulfoton	82677	D	SPE/GCMS <sup>c</sup>	0.021
		EPTC	82668	D	SPE/GCMS <sup>c</sup>	0.0020
		Ethalfuralin	82663	D	SPE/GCMS <sup>c</sup>	0.009
		Ethoprophos	82672	D	SPE/GCMS <sup>c</sup>	0.005
		Fonofos	04095	D	SPE/GCMS <sup>c</sup>	0.0027
		Lindane	39341	D	SPE/GCMS <sup>c</sup>	0.0040
		Linuron	82666	D	SPE/GCMS <sup>c</sup>	0.035
		Malathion	39532	D	SPE/GCMS <sup>c</sup>	0.027
		Metolachlor	39415	D	SPE/GCMS <sup>c</sup>	0.013
		Metribuzin	82630	D	SPE/GCMS <sup>c</sup>	0.006
		Molinate	82671	D	SPE/GCMS <sup>c</sup>	0.0016
		Napropamide	82684	D	SPE/GCMS <sup>c</sup>	0.007
		p,p'-DDE	34653	D	SPE/GCMS <sup>c</sup>	0.0025
		Parathion	39542	D	SPE/GCMS <sup>c</sup>	0.007
		Parathion-methyl	82667	D	SPE/GCMS <sup>c</sup>	0.006
		Pebulate	82669	D	SPE/GCMS <sup>c</sup>	0.0016
		Pendimethalin	82683	D	SPE/GCMS <sup>c</sup>	0.010
		Phorate	82664	D	SPE/GCMS <sup>c</sup>	0.011
		Prometon	04037	D	SPE/GCMS <sup>c</sup>	0.015
		Propachlor	04024	D	SPE/GCMS <sup>c</sup>	0.010
		Propanil	82679	D	SPE/GCMS <sup>c</sup>	0.011
		Propargite	82685	D	SPE/GCMS <sup>c</sup>	0.023
		Propyzamide	82676	D	SPE/GCMS <sup>c</sup>	0.0041
		Simazine	04035	D	SPE/GCMS <sup>c</sup>	0.011
		Tebuthiuron	82670	D	SPE/GCMS <sup>c</sup>	0.016
		Terbacil	82665	D	SPE/GCMS <sup>c</sup>	0.034
		Terbufos	82675	D	SPE/GCMS <sup>c</sup>	0.017
		Terbuthylazine	04022	A	SPE/GCMS <sup>c</sup>	0.1
		Thiobencarb	82681	D	SPE/GCMS <sup>c</sup>	0.0048
		Triallate	82678	D	SPE/GCMS <sup>c</sup>	0.0023
		Trifluralin	82661	D	SPE/GCMS <sup>c</sup>	0.009

**Table 8.** Synthetic organic constituents analyzed by the U.S. Geological Survey National Water Quality Laboratory for the Triangle Area Water Supply Monitoring Project, and the methods used for sample analysis.—Continued

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Class of organic compound	Method reference <sup>a</sup> and notes	Constituent name	Parameter code	Method code	Analytical methods	Reporting level, in µg/L
<b>Pesticides, filtered</b> schedule 2050	O-1131-95 (Werner and others, 1996) collected in the 1994 – 2001 water years	2,4,5-T	39742	B	SPE-HPLC <sup>d</sup>	—
		2,4-D	39732	B	SPE-HPLC <sup>d</sup>	—
		2,4-DB	38746	A	SPE-HPLC <sup>d</sup>	—
		Silvex	39762	B	SPE-HPLC <sup>d</sup>	—
		3-Hydroxycarbofuran	49308	A	SPE-HPLC <sup>d</sup>	—
		4,6-Dinitro-2-methylphenol (DNOC)	49299	A	SPE-HPLC <sup>d</sup>	—
		Acifluorfen	49315	A	SPE-HPLC <sup>d</sup>	—
		Aldicarb	49312	A	SPE-HPLC <sup>d</sup>	—
		Aldicarb sulfone	49313	A	SPE-HPLC <sup>d</sup>	—
		Aldicarb sulfoxide	49314	A	SPE-HPLC <sup>d</sup>	—
		Bentazon	38711	A	SPE-HPLC <sup>d</sup>	—
		Bromacil	704029	A	SPE-HPLC <sup>d</sup>	—
		Bromoxynil	49311	A	SPE-HPLC <sup>d</sup>	—
		Carbaryl	49310	A	SPE-HPLC <sup>d</sup>	—
		Carbofuran	49309	A	SPE-HPLC <sup>d</sup>	—
		Chloramben, methyl ester	49307	A	SPE-HPLC <sup>d</sup>	—
		Chlorothalonil	49306	A	SPE-HPLC <sup>d</sup>	—
		Clopyralid	49305	A	SPE-HPLC <sup>d</sup>	—
		Dacthal monoacid	49304	A	SPE-HPLC <sup>d</sup>	—
		Dicamba	38442	A	SPE-HPLC <sup>d</sup>	—
		Dichlobenil	49303	A	SPE-HPLC <sup>d</sup>	—
		Dichlorprop	49302	A	SPE-HPLC <sup>d</sup>	—
		Dinoseb	49301	A	SPE-HPLC <sup>d</sup>	—
		Diuron	49300	A	SPE-HPLC <sup>d</sup>	—
		Fenuron	49297	A	SPE-HPLC <sup>d</sup>	—
		Fluometuron	38811	A	SPE-HPLC <sup>d</sup>	—
		Linuron	38478	A	SPE-HPLC <sup>d</sup>	—
		MCPA	38482	A	SPE-HPLC <sup>d</sup>	—
		MCPB	38487	A	SPE-HPLC <sup>d</sup>	—
		Methiocarb	38501	A	SPE-HPLC <sup>d</sup>	—
		Methomyl	49296	A	SPE-HPLC <sup>d</sup>	—
		Neburon	49294	A	SPE-HPLC <sup>d</sup>	—
		Norflurazon	49293	A	SPE-HPLC <sup>d</sup>	—
		Oryzalin	49292	A	SPE-HPLC <sup>d</sup>	—
		Oxamyl	38866	A	SPE-HPLC <sup>d</sup>	—
		Picloram	49291	A	SPE-HPLC <sup>d</sup>	—
		Propham	49236	A	SPE-HPLC <sup>d</sup>	—
		Propoxur	38538	A	SPE-HPLC <sup>d</sup>	—
		Triclopyr	39235	A	SPE-HPLC <sup>d</sup>	—

<sup>a</sup>U.S. Geological Survey analytical method reference code.

<sup>b</sup>Solid-phase extraction and GC/MS with selective ion monitoring.

<sup>c</sup>C-18 solid-phase extraction and capillary-column GC/MS.

<sup>d</sup>Carbopak-B solid-phase extraction; high-performance liquid chromatography.